Tappi

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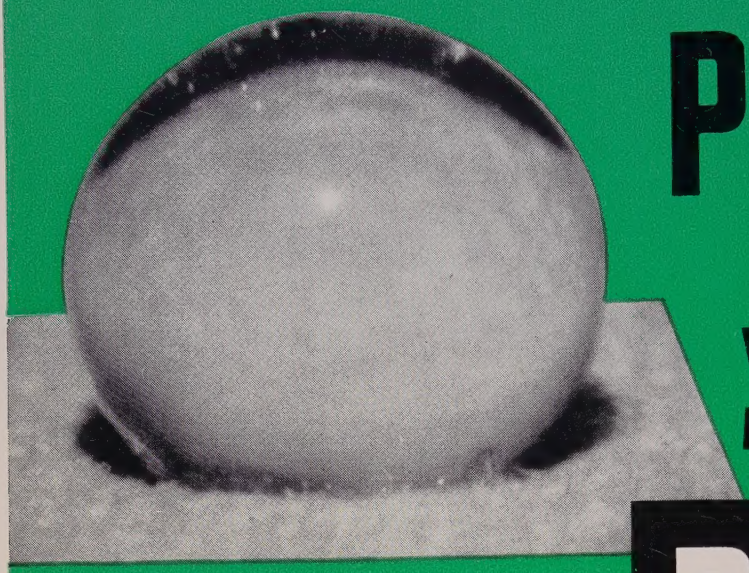
JULY 1952



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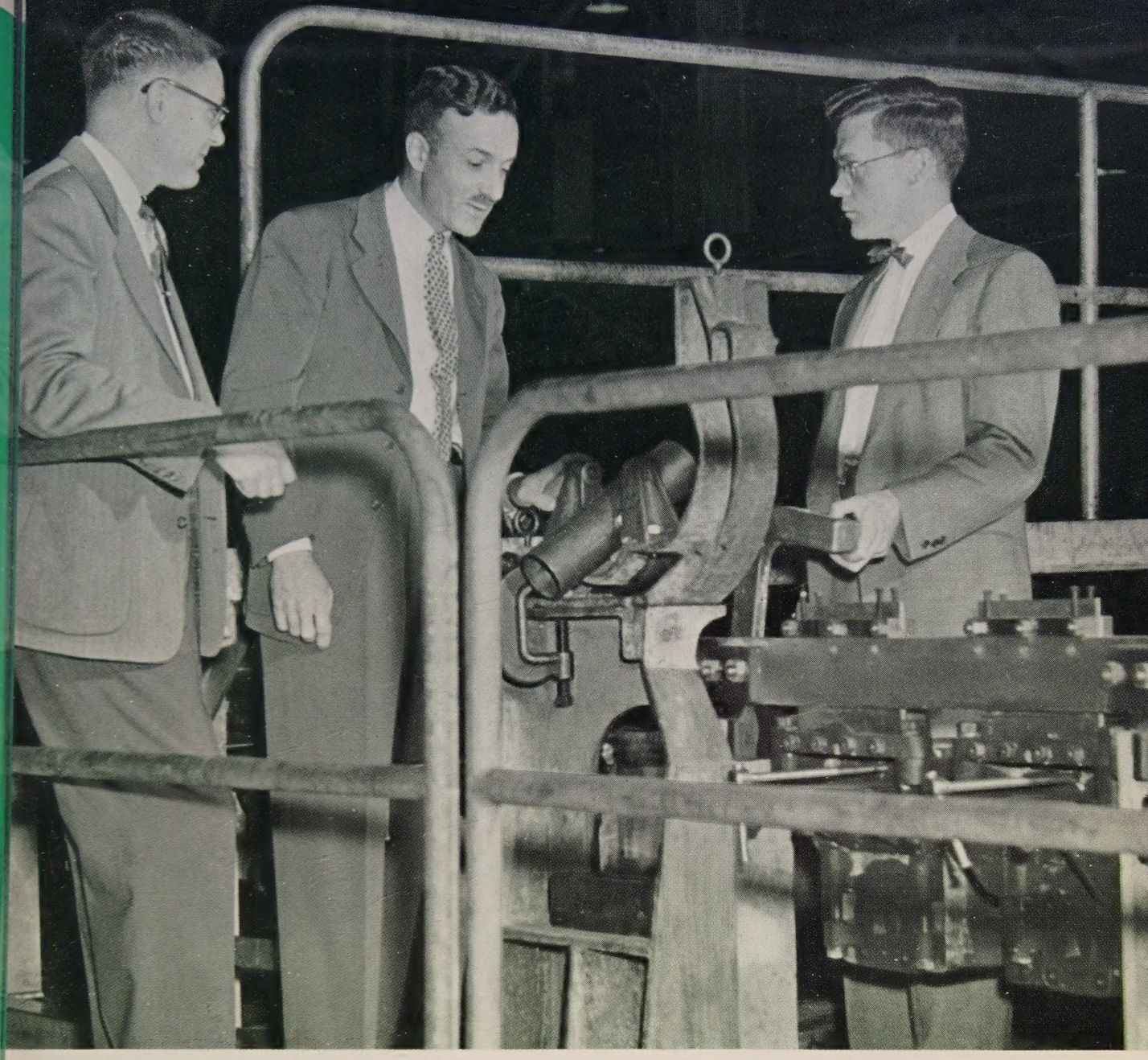

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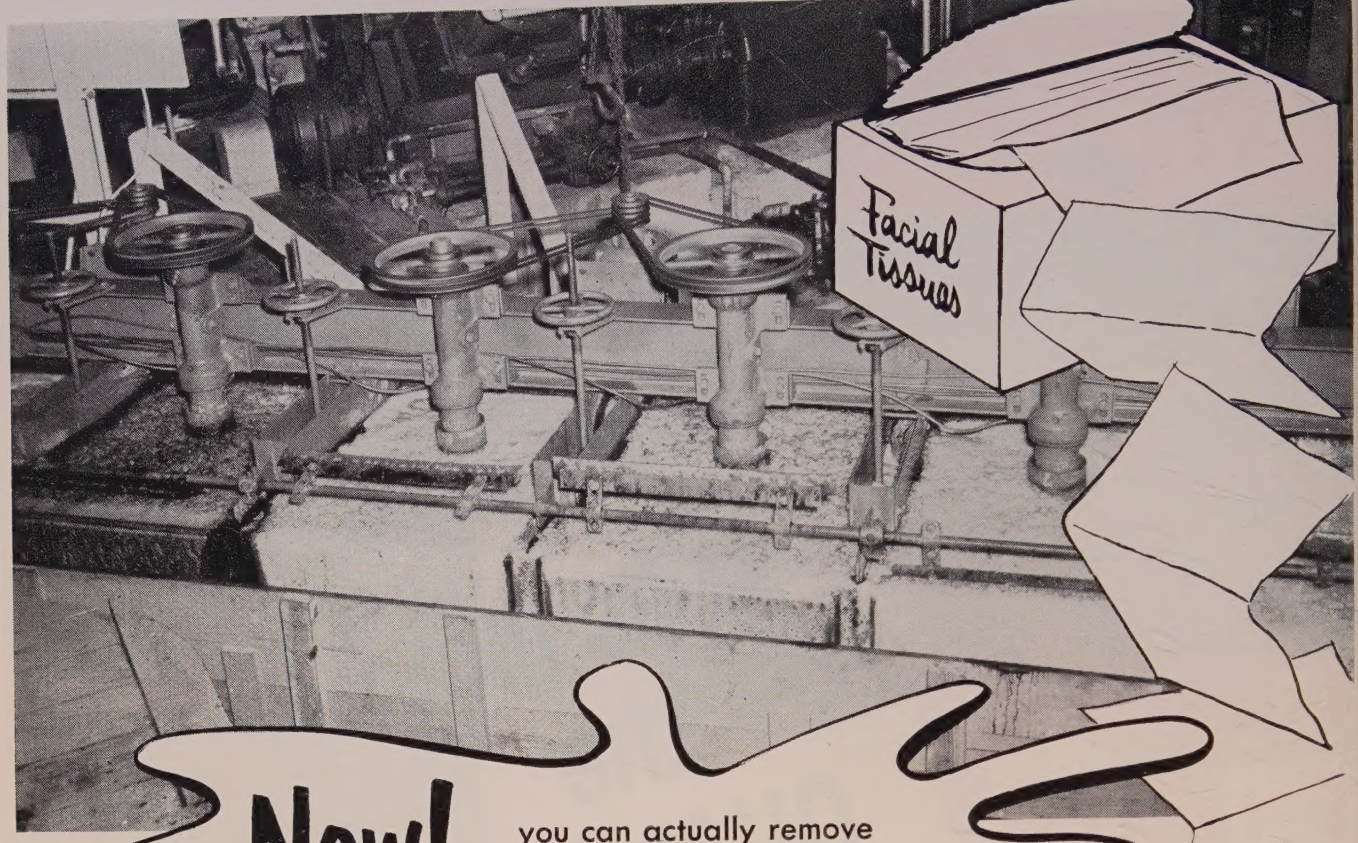

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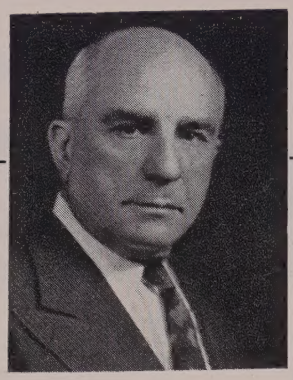

NEW HORIZONS IN PAPER COATING

PETER J. MASSEY

THE steady expansion of demand for our established products and the rapid development and growth of new processes and products make all of us in the field of paper coating keenly aware that there is no other field in the industry which is more important or, indeed, more glamorous than the one in which we are engaged. Today I would like to review the progress which has been made in paper coating during the past few years and to emphasize especially those areas which I believe are the most promising fields for further progress in the industry as well as the most remunerative fields of opportunity for the young technical man of today.

One result of the development of these new fields has been the creation of an ambiguity in the meaning of the term "coated paper." We can all remember when this term was considered the exclusive property of the book paper manufacturer, and denoted a sheet to which clay had been applied for the purpose of imparting better printing characteristics. However, in recent years converters, using modifications of the clay coating techniques and evolving new methods of their own, have been responsible for the production, in very significant volume, of a quite different type of "coated paper."

Perhaps, because of the similarity of production methods, it was natural to use this term to describe the converter's product—the paper-supported functional film. But there is an obvious disadvantage in using the same name to describe two distinct businesses with such dissimilar end products. Therefore, in this paper I will make what I believe to be a useful distinction between the two by referring to the book paper manufacturer's products as *printing coatings* and the converters' products as *functional coatings*.

PRINTING COATINGS

Included in this classification are all of the aqueous dispersed pigment coatings applied on and off the machine to a primary base stock for the purpose of imparting improved printing characteristics to the finished sheet.

The growth in this field since 1933, the year of the first on-the-machine coating operation, has been tremendous, surpassing even the bold estimates made by the most enthusiastic promoters of the machine coating process. The amount of base stock coated by the new method in that first year was 100 tons. In 1950, the latest year for which figures are available, the Bureau of the Census identified 1,020,000 tons o base stock which had been machine coated.

A further illustration of this dramatic expansio is offered by the Census figures which show that durin the three years, 1948, 1949, and 1950, the productio of machine-coated raw stock was increased by 393,00 tons or 63%. During the same period total pape production increased only 13%, and one third of thi total increment was accounted for by the remarkabl expansion of machine coating.

There are two reasons for this achievement. Th first is that machine coating has made it possible fo us to offer to the printer what he wanted—a shee with a plane surface which has improved the quality of his product. And second, the machine coatin process made this sheet available to him at a pric which he could afford. These two elements—improve quality and reduced cost—are the keys to furthe expansion in this field. There is a challenge here and, to the technical man who wishes to accept it, would like to suggest two of the many avenues whic are open to him.

First, intensify present efforts to have coated pape meet all the functional specifications required i planographic printing, both direct and offset methods The technical problems involve the elimination o surface pick and stock splitting in paper used fo multicolor methods which require superimposition o four and five wet ink impressions and improvemen in resistance of the coating surface to prevent con tamination and build-up of foreign material on th printing blanket. The accomplishment of these ob jectives will go a long way to securing increased ton nage from this branch of the graphic arts.

Second, in the rotogravure field, the high fidelity o reproduction of which the intaglio printing process capable can only be achieved with a sheet which sufficiently resilient to permit penetration to the botto of the recessed parts of the plate. As we know, th use of coated paper in this field is limited, at presen by the rigidity of its surface. However, sever latices have been developed recently which show grea promise of being capable of imparting the desire characteristics of greater resiliency and flexibility the coating. These products are now available to th coating technician, and I am confident that a she will soon be developed which fully meets the end-u requirements of the intaglio printer.

The result of these achievements could well be 30% increase in the present machine-coated pap tonnage.

While off-the-machine coating has not matche the extraordinary growth of machine coating, it h enjoyed a market acceptance which could not ha been foreseen ten years ago. It has continued to used for the production of special sheets to meet speci end uses where the volume has not been large enou for the economical machine coating production and h

PETER J. MASSEY, Consultant, River Forest, Ill.

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also met a steadily expanding demand for such products as colored enamels, double-coated papers, household papers, and specialty papers. An off-the-machine product which has created a new market is the coated sheet that, in addition to having good letterpress printing characteristics, is capable of being heat sealed.

FUNCTIONAL COATINGS

The startling developments in functional coatings are so important that one can hardly overemphasize their significance. Unlike the majority of new developments which merely substitute improved products for those which the industry already produces, functional coatings can give the paper industry entirely new products which either have no competition or will compete with and replace the products of the metal, glass, wood, textile, leather, plastics, and other industries. In either case, they will create a new market for paper. Certainly a development which has such revolutionary implications deserves consideration by the industry.

To young engineers and technicians, this sound and promising phase of the paper industry offers outstanding opportunities for a satisfying and rewarding career.

Materials and Methods

The term "functional coatings" has been given to those coatings which impart to the paper a specific function, protective or decorative, which is not characteristic of a paper sheet. A partial list of the substances available for such coatings includes waxes, oils, asphaltic compounds, adhesives, rosins, metallic foils, cellulose esters and ethers, elastomers and other latices, and thermosetting and thermoplastic resins. Using one of the several means at his disposal, the paper technician can apply these materials to paper, which not only acts as an ideal supporting medium, but also furnishes the flexible fibrous constants necessary to meet end-use specifications. Having properly selected his components of paper and coating, he is able to produce a sheet with the chemical and physical characteristics required for almost any conceivable end use.

Waxed and Oiled Papers. The waxed and oiled paper field enjoyed immediate success because it met a definite economic need —the requirement for papers which were water and water vapor resistant. The constant progress and expansion in this field have been due primarily to the advances made in the technology of wax production and the cooperative development by converters and wax producers of nomenclature, instrumentation, and testing procedures. Today, the compounding needs of the converter are met by an extensive variety of waxes having a melting point range from 90 to over 230°F. and a similarly wide range of color, tack, hardness, and plasticity.

Converters have also extended the functional utility of their products by compounding wax with other materials A combination of wax with rubber and other elastomers has been used for cheese wraps and end seals, and rosin-wax and resin-wax compounds have been developed to effect improvements in water resistance and laminating qualities. Ethyl cellulose-wax compounds have chemical and physical properties of considerable merit, but, until their cost can be reduced, their application will be quite limited.

The waxing phase of coating, in common with other converting operations, has been materially affected by the contribution made to it by polyethylene. The lower molecular weights of this resin are fully compatible with wax, and additions of as little as 5 to 10% will improve many of the physical properties of wax coatings. The improved gloss, flexibility, nonscratch surface, and nonblocking and heat-sealing qualities have made the wax-coated product more valuable for previous end uses and have also extended its application to new fields.

The low cost of wax-coated products, achieved through the use of relatively inexpensive materials and high-speed production processes, assures continued success.

In 1950, the consumption of raw stock for waxed paper manufacture was 192,570 tons.

Asphalt Coated Papers. The coating and laminating of paper with asphaltic compounds is one of the oldest coating operations in the paper industry.

The acceptance and continuous expansion of these products have been the result, as in the case of wax coating, of fulfilling the demand for a low-priced water-resistant sheet. Furthermore, recent improvements in the physical properties of asphaltic materials and the development of such additives as asphaltic resins which raise the melting point of the compound have considerably expanded the end uses of asphalt-coated papers.

The Bureau of the Census reports that in 1950, 176,270 tons of raw stock were consumed in the production of these papers.

Solvent Resin Coatings. Solvent coating of paper has had a steady growth and expansion. To this coating process must be given the credit for the introduction of paper-supported thermoplastic resins to the paper industry.

Solvent coating and/or saturating of paper with thermosetting resins has had constant expansion primarily because it has been successful in meeting the products of the metal, wood, textile, and leather industries in free competition. Its future success is assured by the constant improvements in raw stocks and resins plus improved manufacturing techniques. The possibilities of substituting aqueous dispersions of the various thermosetting resins for the solvent resin compounds is being explored with some indications of progress. If this could be successfully accomplished, it would materially lower the costs and increase the speed of production.

The statistics from the 1950 Census Report indicated that the raw stock consumed was 49,930 tons.

Solvent coating of paper with thermoplastic resins has been successful. The first paper-supported polyethylene film was produced by this process. I have no statistics as to the raw stock consumption in this field.

Rubber and Other Elastomer Latices. The great bulk of this operation, primarily a saturating process, is performed off the paper machine. Retentions of up to 100% of the elastomer relative to the basis weight of the raw stock are common practice in the production of sheets which meet the requirements of the users of these materials.

Considerable attention and effort have been directed to the production of such papers through both beater

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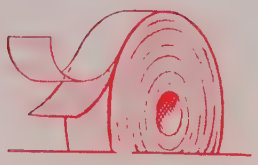
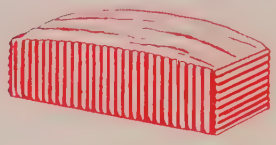


For rubber-separator papers, pressure-sensitive tapes and labels, "Quilon" makes possible easy stripping.



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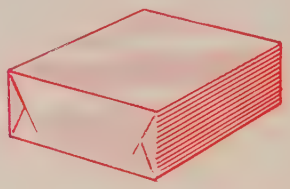

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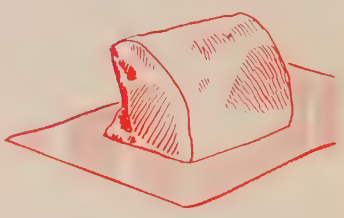

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addition and size tub on the machine methods, and I have had reports and samples of products with an elastomer content as high as 35%. However, while there is some demand for a product in this range, the major demand is for products having a retention of 70 to 100%.

Even if the 100% goal were to be achieved on the machine, it would not materially affect the production of the off-the-machine operators, for the specialized production orders which they typically receive are too small for economical production on a paper machine. Machine operations would be directed to the production of sheets for end uses where a lower percentage of latex is specified and for large volume orders which could be produced at less cost than the off-the-machine product.

The specifications for an elastomer-latex sheet are determined by the end-use requirements. Many end uses demand aging and color stability and light and heat resistance in addition to the normal intrinsic tensile, stretch, fold, and elasticity properties.

While it is known that the products of this activity have been successfully marketed in competition with the products of the leather and textile industries, production statistics are not available.

Thermoplastic Coatings. The interest of the paper industry in thermoplastics generally was purely academic until the advent of polyethylene. The intriguing chemical and physical properties of the higher molecular weights of this resin encouraged increased efforts to find ways and means of applying high polymers to paper. All of these efforts have occurred since the last war, for, while the resin has been produced in the United States since 1942, the entire wartime production was used for insulation purposes by the military. Thus, it was not until 1946 that large quantities of polyethylene became available for civilian use.

With the end of hostilities new outlets were sought for the expanding production of the chemical companies.

One of the two leading producers had had a long background in the manufacture of extruded films, and consequently its initial efforts were directed to the production of a polyethylene film. These efforts were immediately successful, but the opportunities for the distribution of the unsupported film were found to be considerably restricted. It was the paper-supported film which had the great market potential.

The first paper-supported film was produced by laminating the extruded film to a base sheet. But the economics of this operation severely limited the full possibilities known to exist for such a sheet, and solvent coaters, who could produce a supported film in one operation, were quick to enter their product in competition with that of the laminators. This pioneering effort furnished the actual evidence of the value of a paper-supported polyethylene film as a packaging material and established the factual data which were used to determine the size of its potential market. The results were so encouraging that the search for an even more economical means of production was further stimulated.

The leading producer of unsupported polyethylene film drew upon its experiences and know-how to evolve a unit which would laminate the extruded film to a paper web as an integral part of the film-forming operation and made this process available to the industry without restrictions of any kind. The process was adopted by a large paper manufacturer whose success has led other companies to install similar equipment.

Simultaneously with this development, equipment was evolved which produced the same end product by the hot melt processing principle, wherein a melter and roll applying means, provide for high-speed production.

At present, to my knowledge, these are the only nonsolvent means of producing a supporting film of polyethylene and other thermoplastic resins.

That all of these developments have been accomplished within the few years since polyethylene was first made available for civilian use indicates the impact which thermosetting resins have had upon the industry. Unfortunately, I can give you no authoritative tonnage figures on either the resin or raw stock consumed by this activity.

This new phase of paper coating has caused the industry to look to TAPPI for answers to the many problems it has raised. At present we are forced to grope blindly, adapting these resins to our production processes by cut-and-try methods. Basic research and application of the research results to our production processes are needed if we are to have the facts required for intelligent application and use of the many resins now available to us.

Our present sistuation with respect to the high polymer resins is very similar to our situation in the early days of machine coating. We formulated coating compositions and produced marketable coated sheets by cut-and-try methods, but it was not until we had learned to evaluate the rheological properties of our coating compositions that we were able to select the right materials for the particular process, accurately predict their behavior on the machine, and know that the resulting product would be satisfactory for a specific end use. Without the instrumentation and tests which made available numerical values of the non-Newtonian properties of our coating compositions, we could not possibly have achieved the present successful accomplishment.

In coating with high polymer resins, we are dealing with materials which are composed of long-chain molecules, are solids at ordinary temperatures, and have very high "viscosities." The viscosities of high molecular weight polyethylene, Saran, vinyl copolymers, cellulose esters and ethers, and S-50 are much higher than we have encountered in dealing with other coating materials and are obviously greater than anything we have met in dealing with clay coatings.

Were these thermoplastics simple Newtonian fluids, a knowledge of their viscosities at their technical extrusion temperatures would make the problem of determining their behavior under varying shear stresses relatively simple. Unfortunately, however, these materials are non-Newtonian and thus the rate of deformation under a certain stress and temperature will not correlate with a single point viscosity determination. Furthermore, the properties of the materials with which we are dealing are determined by a complex of rheological factors of which viscosity is only one.

At present, our only knowledge of the rheological properties of these materials is limited to the infor-

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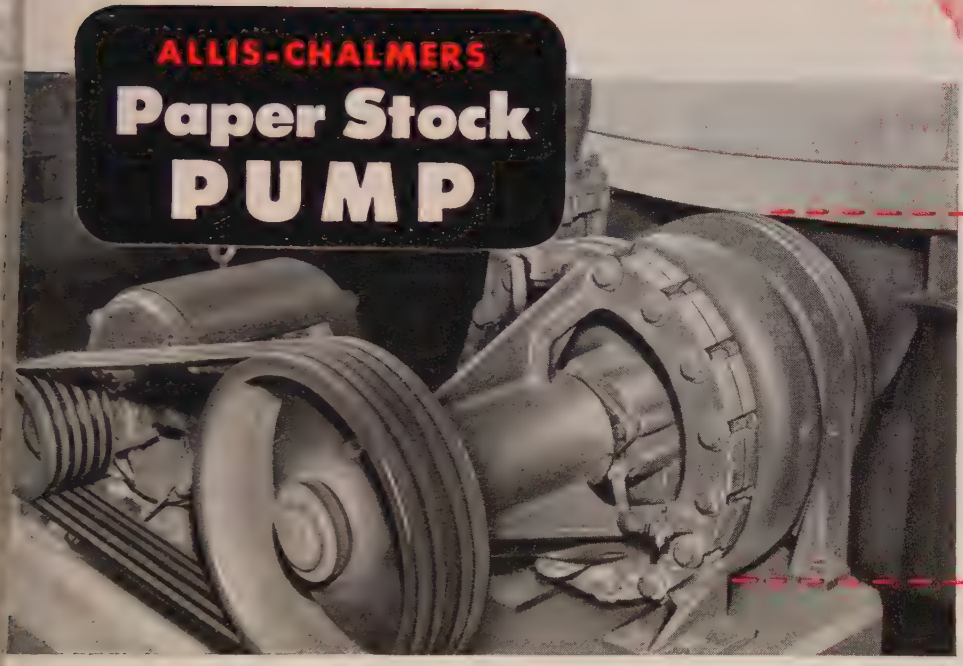

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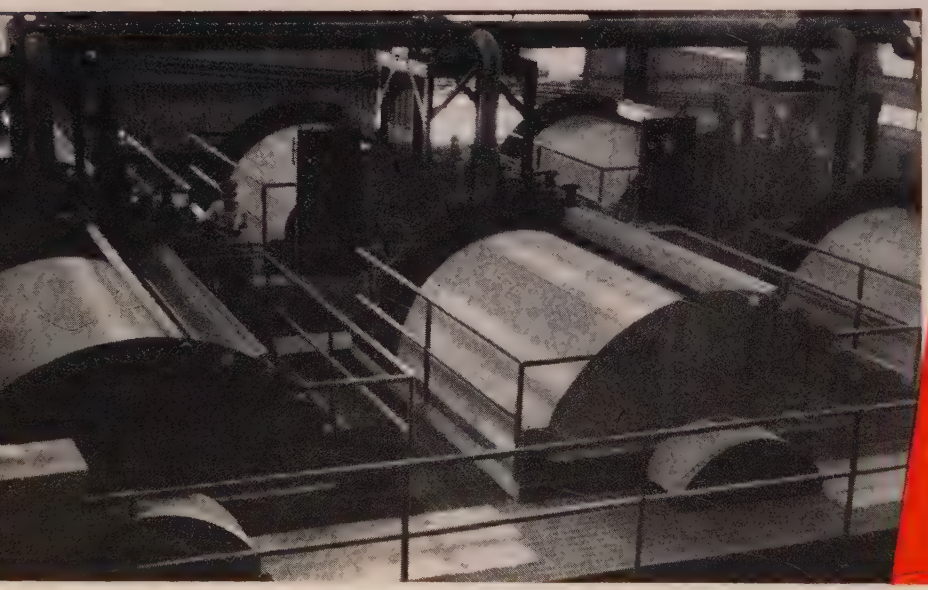
mation which we can obtain from our most modern plastometers. All of those which are capable of subjecting high polymer thermoplastics to high rates of shear are of the extrusion type which extrude at the rate of an inch or two per minute and give single point viscosity values at a single arbitrary temperature and pressure. Obviously, while the results of such tests would be of value for quality checks on various shipments of some one resin, they are hardly applicable for measuring the resin's behavior on processing equipment operating at 400 f.p.m., and creating temperatures and shear stresses considerably greater than those generated by the plastometers.

The further exploitation of paper-supported thermoplastic films will be infinitely advanced by the development of instruments and tests which will cover the extensive ranges of shear stresses and temperature encountered in operation, and will measure the elastic properties of the thermoplastic and the conditions of thermal decomposition.

In this presentation, I have avoided any discussion of the base material, paper, which is our main interest. That paper is the foundation and most important element in any type of coating is true, and the building of a sheet having the necessary physical and chemical constants for any given process or end use is part of your daily tasks and problems. The reason for omitting this subject is that it is too important to be covered adequately in a paper of this length, and I suggest that the committee place this important subject on the agenda for a future meeting.

I believe that the opportunities which the field of paper coating offers the technical man of today cannot be surpassed by any other field of endeavor, and I am sure there is no field more challenging. Our expansion has been due in large part to the contributions of TAPPI and I trust that the few problems touched upon today will be taken as a real challenge by our younger members, thereby assuring, indeed, new and brighter horizons in paper coating.

PRESENTED at the Third Coating Conference of the Technical Association of the Pulp and Paper Industry, Chicago, Ill., May 5–8, 1952.



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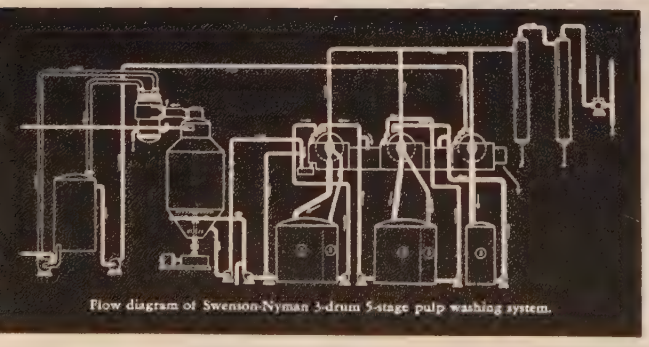

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Picture of Gurley Densometer.
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The Future Requirements of Starch from the Standpoint of the Paper Coater

F. H. FROST

Those of you who have been in the coating field for 20 years or longer will recall how meager our knowledge was of the general subject of coating in the twenties and early thirties. Assuredly we have made much progress since that time; progress in which we can, I believe, take some justifiable pride even though we will all admit there is much good work to be done and many answers we do not now possess.

I am particularly impressed with the plan of this meeting. T. J. Schoch, an expert in the fundamental chemistry of starch, will discuss the possible relationship between molecular structure and behavior and our everyday problems and concepts. I have agreed to attempt to point out observations we have made on the relationship between variables in our coatings and Mr. Schoch will attempt to relate these to fundamental starch chemistry. Obviously he has the difficult job since we all know it is easy to ask questions.

I am impressed because the real lead-off speaker of this meeting has been working and studying very closely to the realm of pure science. It is certainly a credit to the starch producers that they have the vision to support such work and a credit to your program committee that they should invite Mr. Schoch to start off this conference. If in the last 20 years we have learned that nearly all practical progress rests upon the foundation of fundamental science, and we must support such work, we have indeed matured; the field of coating is being transferred, as it should, from an art to a science.

The first question I should like to raise, we might call "water retention of coatings."

If we sprinkle a dry indicator, such as potassium permanganate, upon an unsized lightweight sheet of paper such as filter paper, and drop the square of paper upon water, the penetration of water through the sheet is so fast we cannot accurately measure the rate of penetration. This is a very simple, easy test.

If we next perform the same test on a starch-clay-water coating—say at 45% solids with 15 parts of starch to 100 dry clay—even though the coating is very fluid, the rate of water penetration is enormously reduced, 25 sec. rather than a fraction of a second may be required.

Does this phenomenon of water retention, which highly fluid starch coatings exhibit, relate to the structure of the starch molecule?

Personally I think it does and if so we have a good question for Mr. Schoch.

If water retention is fundamental it should correlate with changes that starch undergoes in processing, coating formulations, and in basic results. Figures have been prepared showing some of these relationships.

Figure 1 shows the changes in water retention which take place during a dextrinization. In all cases 100 parts clay, 15 parts starch, at 45% solids have been used unless otherwise noted.

F. H. Frost, Research Manager, S. D. Warren Co., Cumberland Mills, Me.

Note that water retention and time of dextrinization indicated by ten time stages correlate with remarkable accuracy. As a matter of fact it appears water retention could be used as a control test for degree of dextrinization. This to me suggests a fundamental relationship.

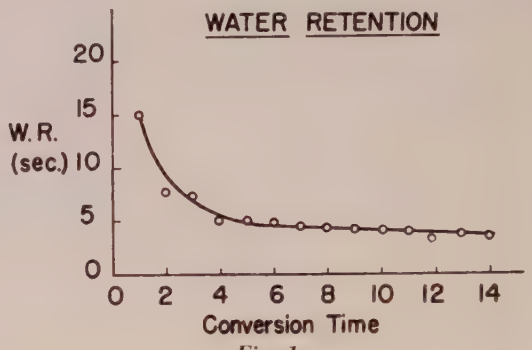

Fig. 1

Figure 2 shows another relationship. In this case, using the same coating proportions, we show the relationship between Dennison wax test (made on coatings applied to a body stock which is very hard and exhibits great resistance to fiber pick) and water retention. The water retention was varied by using starches converted by different methods and to different degrees. As you note again we have an excellent relationship which in effect means it is possible to predict a wax test by measuring water retention before a sheet is coated.

Figure 3 shows the relationship between water retention and amount of starch used. As we increase the amount of starch the water retention increases.

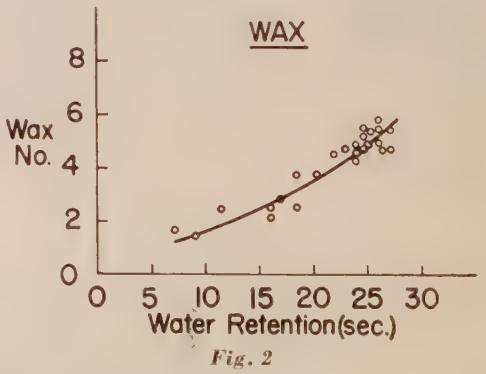

Fig. 2

Figure 4 shows the relationship between the amount of starch and wax test. Hence, increasing the amount of starch produces an increase in both the wax test and the water retention. This factor, therefore, in turn produces a correlation between the wax test and water retention as a comparison between Figs. 3 and 4 will illustrate.

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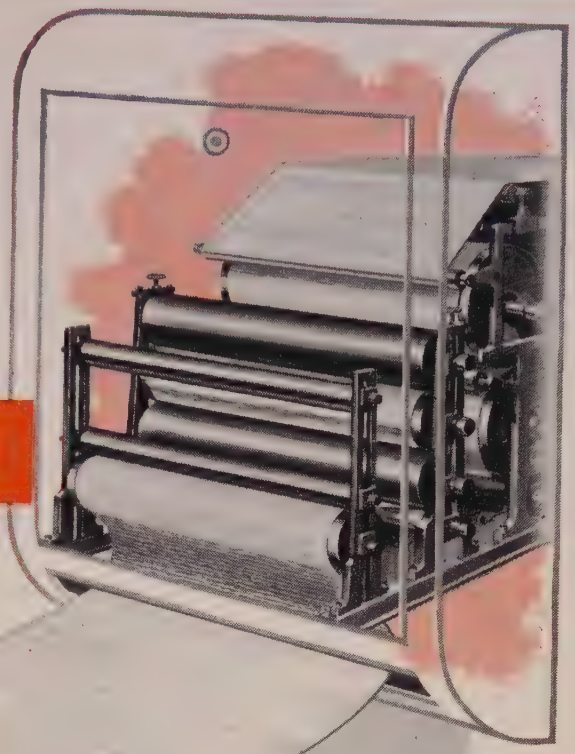

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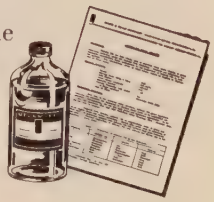

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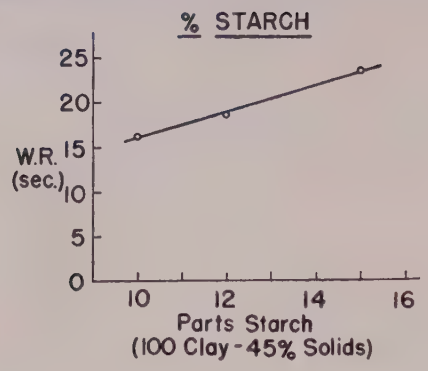

Fig. 3

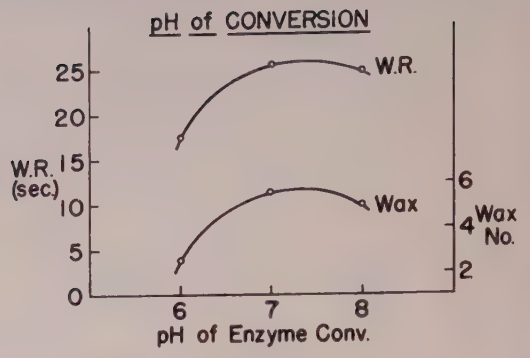

Fig. 6

Figure 5 shows the relationship between per cent solids of a coating and water retention. We know an increase in solids gives us an increase in pick resistance so it is not surprising to find the relationship illustrated in Fig. 5.

Figure 6 shows two curves for wax test and water retention versus the pH of an enzyme conversion—again we find an excellent correlation along more or less expected lines.

Figure 7 shows the correlation between wax test and water retention with the amount of enzyme used in a conversion. The correlation is excellent.

Figure 8 gives wax tests and water retention results versus the end-point temperature of a cook of chlorinated starch.

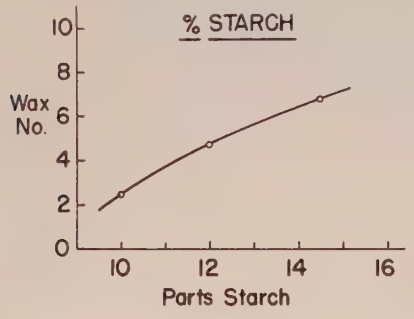

Fig. 4

Increasing the end-point temperature of the cook apparently affects the starch dispersion in a vital manner.

It appears to me the above results suggest strongly that the variable of water retention, which correlates with other important coating characteristics, is tied in directly with the molecular structure of starch. If Mr. Schoch agrees with this conclusion, we ask him how such a relationship can be explained.

When a body stock is to be coated one of our chief objectives is to form a layer of coating on the surface of the body stock. We can accomplish this only if a major percentage of the coating applied remains on the surface.

Figure 9 shows the relationship between the per cent solids of the coating composition or mixture and the percentage of coating to the total weight of coating applied which remains on the surface of a body stock.

We all know that one of the most important effects we must obtain when we coat a sheet of body stock is to obtain a layer of the coating material which is continuous on the surface and which buries the fibers. If we fail to get coating holdup we get the effect of an impregnated sheet. Our continual striving for higher and higher solids is a reflection of the desire to obtain coating holdup.

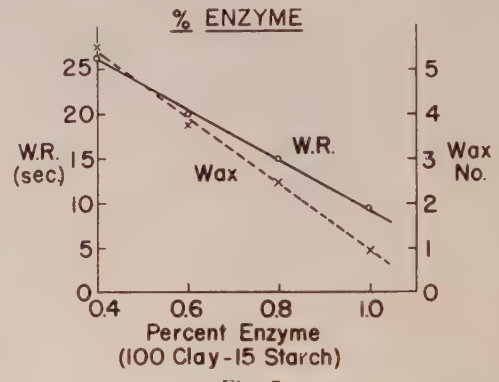

Fig. 7

Now we know that as solids increase both apparent viscosity and water retention also increase but it is questionable how much effect either of these factors has if studied independently of per cent solids. I have seen results which indicate that high viscosity, on occasion at least, can produce a poorer holdup of coating. If we had a new variable which was independent of per cent solids and which would aid us in holding a coating up on the surface of a body stock it would be most valuable. I think it a fair question to ask

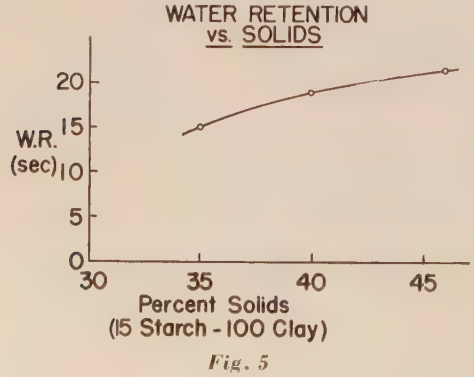

Fig. 5

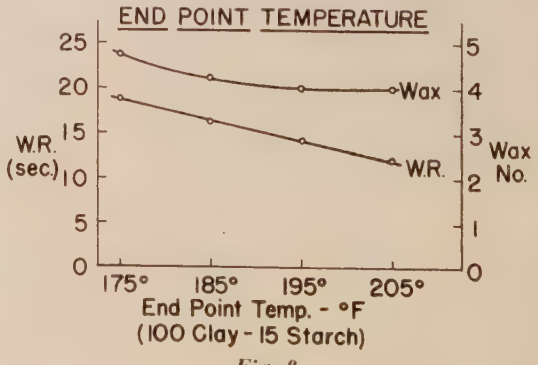

Fig. 8

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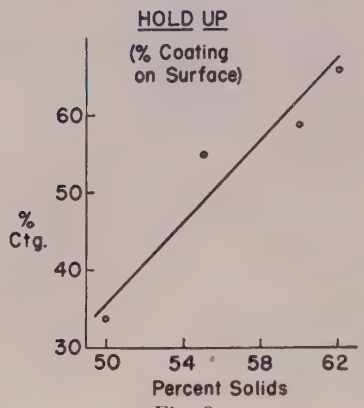


Fig. 9

if there is a possibility that such a new variable will be unearthed as a result of fundamental studies on the structure of starch. Have we missed a characteristic capable of being varied in starch coatings which might be important to this question of coating holdup? I hope we will have some suggestions on this point. Hence that is question No. 2.

Question No. 3 relates to the effectiveness of starch as a pigment binder or, as we loosely say, its adhesive strength. For years we have accepted the fact that starch is weaker or

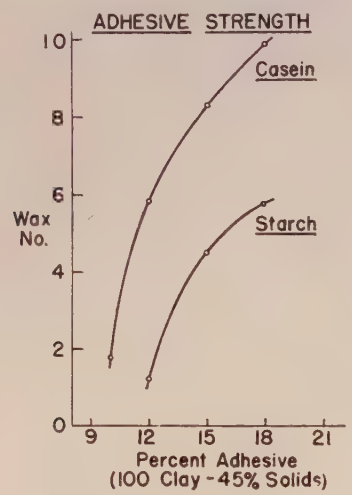


Fig. 10

has poorer adhesive binding power than casein. Why? What is the fundamental reason? This relationship is shown in Fig. 10.

We should add, however, at this point that adhesive power, *per se*, as measured by the Dennison wax test or a controlled printing test, is not enough. On certain specialized body stocks satisfactory binding may be obtained with as low as 11

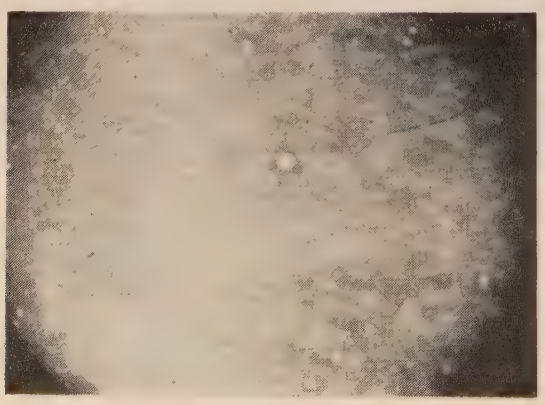


Fig. 11

Fig. 12

parts of starch to 100 clay yet a satisfactory surface is not obtained. In such cases, unlike a casein coating of 11 casein to 100 clay, we find the starch coating extremely sensitive to scratching—it is very fragile and will not withstand normal handling on printing equipment. Again, we ask, why?

Question No. 4 relates to wet adhesive of a coating. In machine coating which involves application of the coating via a squeeze roll system, the wet coating as it emerges from the

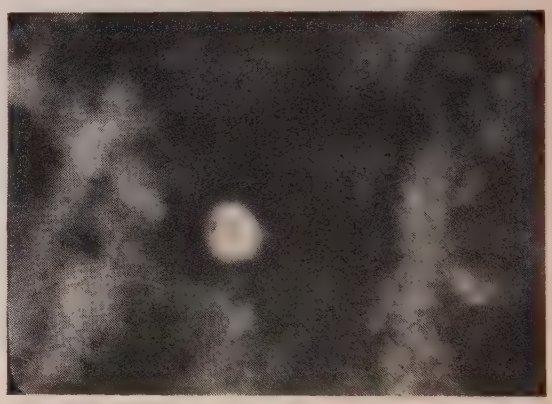


Fig. 13

nip must break away from the applicator roll. If extensive necking occurs a bad pattern results. The property of wet adherence we might well imagine is tied into fundamental starch chemistry.

Question No. 5 relates to apparent viscosity in general. Viscosity or plasticity is a factor of importance in all coating operations. Without asking Mr. Schoch to go into extensive detail I do think it will be most helpful if he will give us his general mental picture of the relation between apparent

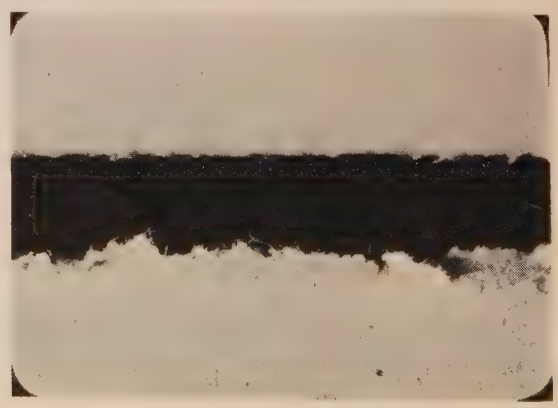
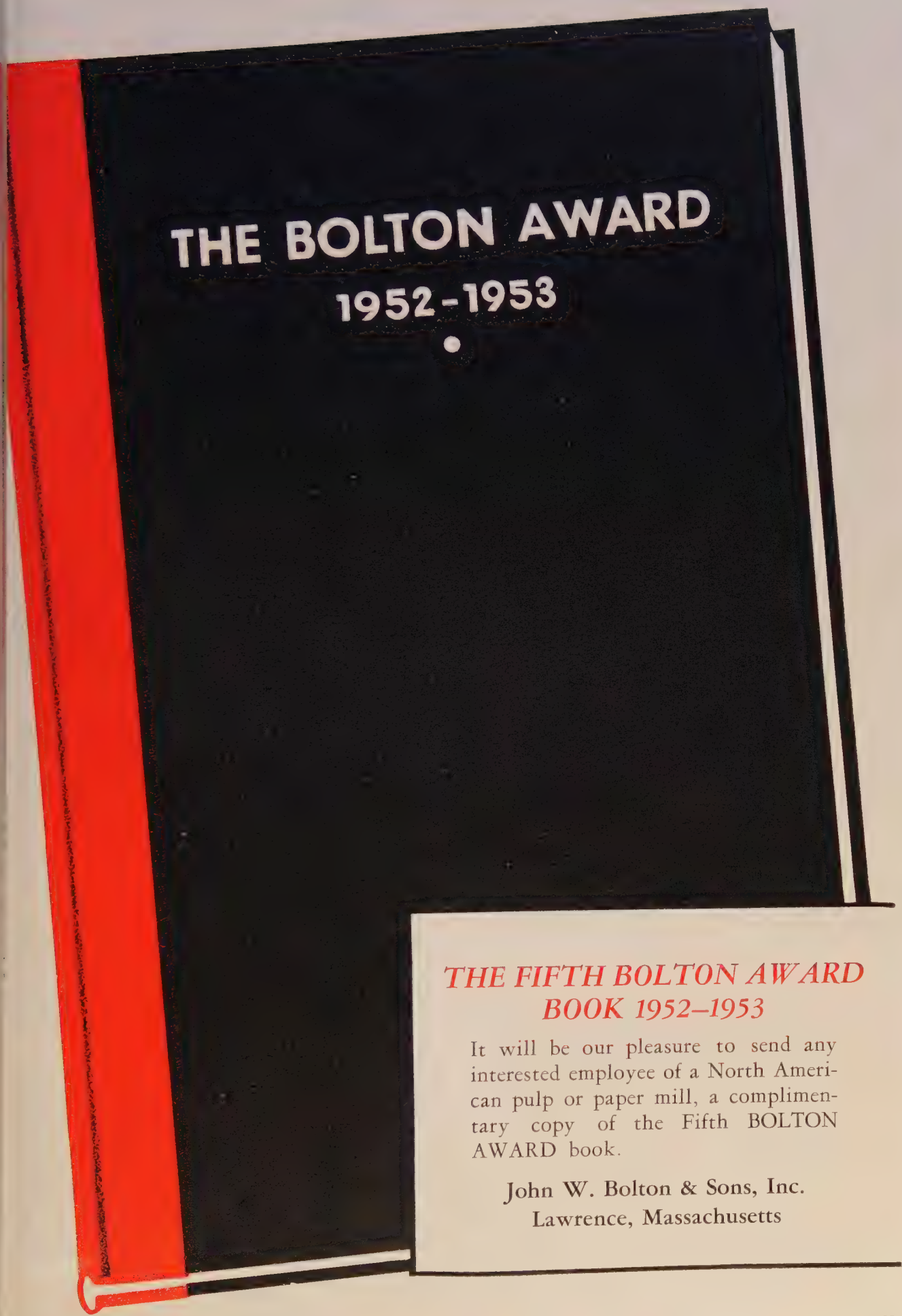


Fig. 14



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viscosity and the fundamental nature of various types of starches. Possibly his remarks on this question will stimulate a worth-while discussion later.

I am hopeful that question No. 6 will produce some lively debate. Let's call No. 6 "The Case of the Undisorganized Starch Granule."

In many instances, especially in enzyme conversions, a portion of the starch fails to go into or remain in a properly dispersed state. When such a situation arises and such a starch is used in a coating, transparent spots develop in the coating. These spots interfere seriously with printing quality. If the starch is centrifuged these undisorganized granules, or whatever they are, are removed. Figure 11 is a photograph taken by transmitted light through a coating and shows these spots. Figure 12 is an identical coating except the starch was centrifuged and the undisorganized granules first removed. Figure 13 is a photograph of a single spot.

Just to prove the point, if we stain the sediment removed by the centrifuge, and mix it back in a coating, these spots will reappear and all will be stained or colored.

It seems to me a must that the starch industry tackle this problem with their heavy artillery at once and give us a decisive solution. So we ask, how come?

I think from time to time we have all been plagued with "calender dust" especially from starch coatings. I have heard a good many diverse theories as to the cause of this condition and if there are some ideas on this point I am sure they will be appreciated.

When this situation develops, a fine dust gradually accumulates on the steel rolls of the calender and, as it builds up, the finish of the paper is seriously impaired.

So question No. 7 is: does this dusting problem tie into the chemistry of starch?

Question No. 8 involves folding, not the Schopper or M.I.T. fold of paper, but the smoothness of the edge of a broadside as folded on say a Cleveland folder. Figure 14 shows a smooth and rough fold. Both starch and casein coatings tend to crack and give a jagged edge especially if folded at low humidities. Synthetic elastomeric adhesives produce superior results. Since in folding, the outside surface is severely pulled and is asked to conform to a longer distance, it has been my opinion that the problem relates to the elasticity of the adhesive used as the binder. If so, what are the possibilities that the fundamental research workers can come up with starches which possess increased elastomeric properties?

Question No. 9 involves enzyme conversion. I think we would all be interested in the latest up-to-the-minute theory in regard to enzyme conversion in general and anything in particular as to new variables that affect a conversion you might care to discuss.

Question No. 10 and the last one is this. In some labeling operations it is essential that the starch coating is reasonably insoluble in water. We appreciate that it is possible to insolubilize starch coatings and to do a practical satisfactory job but the usual methods involve curing at a pH below 7. Is the starch industry aware of the work that has been done at the Lithographic Technical Foundation which shows that the pH of a coating has a major effect on the rate of ink drying and that a pH on the acid side produces a hazardous situation? We need methods which will give cures on the alkaline side. Is this a practical possibility?

These are the ten questions. I appreciate that these are a selection from many that could be asked and that others will be asked as the meeting progresses.

In closing may I again say the coating industry appreciates the fundamental research that is being done in the field of starch coating and that we all eagerly await the solid foundation the starch industry is building under our applied science.

Fundamental Developments in Starch for Paper Coating

THOMAS JOHN SCHOCH

Mr. Frost has posed ten problems of entirely practical character. He has described certain desired qualities of starch which we have not entirely achieved, accenting those instances where there is room for improvement or for new and different starch modifications more suited to his specific needs. And we particularly welcome that sort of discriminate comment.

However, this matter of new and different starches is not quite as simple as it may seem. In the earlier days of the starch industry, progress was made by the simple expedient of treating starch with chemical X, heating the mixture for several hours, and discovering a novel and useful result. Most of our dextrins, thin-boiling and oxidized starches, were developed in this Edisonian fashion. But the easy and ordinary and inexpensive things have been discovered, and from here on the going becomes tougher, further complicated by the fact that you starch users are becoming more insistent on special characteristics. The starch industry realized some 15 years ago that it could not continue to develop solely by pragmatic means, that it must use every scientific approach available, particularly a sound working knowledge of what the starch molecules look like and how they behave under any given set of conditions. The starch technologist of 30 years ago didn't give a hoot about molecules. But how frequently of late years have I heard my very practical-minded associates make some comment which revealed that they were trying to grasp the fundamental character of a particular problem, trying to outguess the molecules. And in 15 years we have made substantial progress, not enough to solve all your problems by a turn of the wrist, but enough to realize dimly that there is some sort of orderly scientific pattern in the behavior of starch. So when your starch supplier seems slow to solve some special problem, please remember that he is trying his level best, utilizing his practical know-how, plus his scientific reasoning, plus a lot of sheer crystal-ball gazing.

I have said that there seems to be an orderly pattern in the behavior of starch molecules. In this paper I would like to discuss in detail what we think this pattern is, how it helps to explain both the desirable and undesirable characteristics of various modified starches and dextrins, and finally what we hope to do in a practical way with these concepts in the future. The way in which a modified starch behaves in a clay coating is determined by such factors as the size and shape of the starch molecules and the attractive forces between them. Even the organization of the native starch granule has an influence, since the starch substance retains the memory of its original native habitat long after it has been pasted and liquefied with enzymes. So I propose first to develop certain pertinent phases of blue-sky starch chemistry, without any mention of clay coatings. I promise then to prove the relevancy of this material to your own interests.

Basically, starch is a polymer of glucose units, hitched together like cars in a train, very similar to the cellulose molecule with which most of you are familiar. But there are a number of important differences from cellulose. In the first place, the glucose units of cellulose are hitched together by so-called beta-1,4 linkages, simply meaning that the first carbon atom of one glucose is coupled to the fourth carbon of the preceding unit through an oxygen bridge on one side of the glucose, as shown in Fig. 1. From space considerations, we find that this gives a rigid rodlike molecule of high structural strength, eminently suited as building material for a cotton thread or for the fibers in a piece of paper. Starch, on the other hand,

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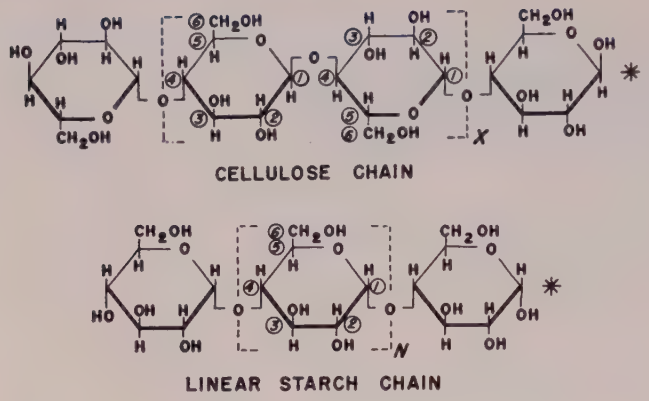


Fig. 1. Polymeric chain structures of cellulose and starch

has alpha-1,4 couplings, with the oxygen bridge on the opposite side of the glucose unit. This imparts a flexibility to the molecule, permitting it to twist and coil and kink like well-cooked spaghetti.

Both cellulose and starch are high polymers heavily loaded with free hydroxyl groups. This imparts a property which both substances have in common, the tendency of the molecules to attract one another and to line up in parallelwise association. This is actually an attractive force between hydroxyl groups, the same sort of thing which makes molecules of water cling to one another, variously termed hydrogen bonding, intermolecular association, or molcohesion. The result is a sort of zipper action—when two or more molecules come into contact, they are zippered together into a tight orderly lattice which the x-ray indicates is actually a crystalline organization. When this happens in a starch system, we give it a name with which many of you are familiar—*retrogradation*. It shows up in the slow precipitation of insoluble starch substance from the dilute solution of starch indicator standing on the laboratory shelf. Under these circumstances, the starch molecules slowly line up and zipper together into aggregates which are just too large to remain in suspension. If the starch system is more concentrated, the closer proximity of the molecules will produce more rapid association without permitting time for maximum orientation. This may give rise to the situation depicted in Fig. 2, whereby we obtain an interlacing network extending throughout the system to give a gel structure. This sort of thing is fine if we want a cornstarch pudding—it may become an unholy nuisance if it occurs in the size kettle of a textile mill.

There is another important difference between starch and cellulose. Cellulose consists entirely of linear chain molecules, while most starches contain two types of polymeric molecules, one a linear chain of perhaps 500 to 1000 glucose units, the other a branched type of structure (Fig. 3). This branching is accomplished through side linkages on carbon no. 6, to build up a huge treelike molecule consisting of some 100 to 200 short linear branches, each of which is 25 to 30 glucose units in length. The proportion of these fractions varies from one starch to another, though it is surprisingly consistent within any given species. Thus cornstarch contains 28 to 29% of linear component, potato starch 20%, and tapioca 17%, the balance in each case being branched fraction. The genetic variety of corn known as waxy maize yields a starch which (when pure) contains absolutely no linear fraction. At the other extreme, the starches of common wrinkled-seeded garden peas and of certain sweet corn types may contain 65 to 85% of linear fraction.

Suppose we consider the matter of retrogradation in some detail, since its occurrence and prevention are of vital importance to every user of starch. And by retrogradation, I mean the tendency of linear starch molecules or segments of molecules to associate in parallelwise fashion, to give an insoluble gel or an insoluble precipitate. Let us first consider potato starch which is not too prone to retrograde. Its pastes are clear and somewhat slimy with little inclination to become opaque or to precipitate on standing. There are two reasons for this stability. In the first place, potato starch contains a relatively lower proportion of linear fraction than do the cereal starches. However, if we isolate the pure linear fraction from potato starch, we find that even by itself it does not readily retrograde. Its solutions show less tendency to gel or to precipitate than do solutions of the linear cornstarch fraction. One plausible theory proposed by the polymer chemists is that the linear fraction of potato starch is some 1000 glucose units in length, just too long to retrograde. Such a ponderous molecule might well show a molecular inertia, a slothfulness to untwine itself and move into proximity with other molecules. In support of this theory, we find that if the linear potato fraction is acid-hydrolyzed into two or three shorter lengths, these fragments are much more inclined to aggregate and precipitate.

Next, let us consider cornstarch, which is known to give strong firm gels and somewhat opaque pastes. It has a relatively higher content of linear fraction—some 28 to 29%. The best estimate of chain length of this linear fraction is about 400 to 500 glucose units, and this length seems just about optimal for gel formation. The linear molecules are long enough to show some inertia of movement, but active enough to permit partial zippering, thus giving the interlacing network of a gel. If we prepare a hot 5% solution of the corn linear fraction and allow this to cool to room temperature, it immediately sets up to a rigid gel, so firm and elastic that it will bounce on the floor. Moreover, this gel represents a permanent retrogradation, since it cannot be reliquefied by heating.

Let us, in turn, hydrolyze the linear cornstarch fraction slightly by treatment with acid, just enough to split it into two or three shorter fragments. We find that it loses its ability to give a gel, but the tendency to give an insoluble precipitate is markedly increased. Hence we may presume that the shorter linear molecules of perhaps 100 to 200 glucose units have greater mobility, more freedom to orient quickly into discrete particles. As we hydrolyze still further, down to a chain length of 20 to 30 glucose units, the linear substance finally loses its inclination to precipitate from solution. Of course, in this process of progressive hydrolytic fragmentation, we have lost many of the useful colloidal properties of big molecules, such as viscosity, emulsifying, and protective colloid action. And if we conduct this hydrolysis on the whole starch, the branched fraction is fragmented right along with the linear. So by the time we have whittled the linear fraction down to a point where it won't retrograde, the starch wouldn't be of much use to your industry.

The branched starch fraction can likewise be fitted into this scheme of retrogradation. It has an enormously large

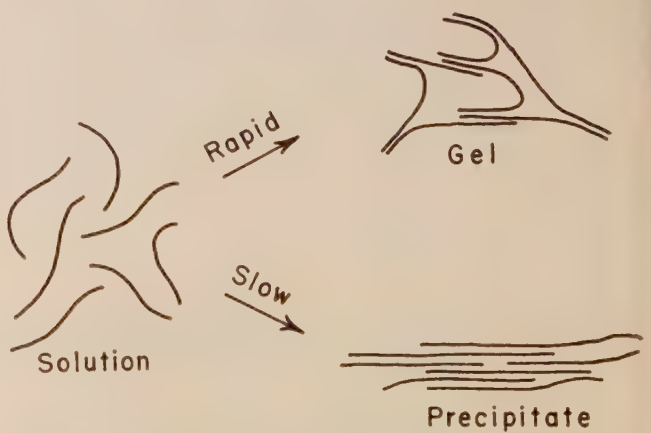
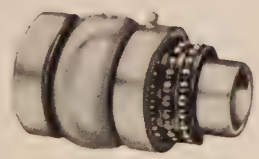


Fig. 2. Mechanisms of retrogradation of the linear starch fraction

pulling together...



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three-dimensional molecule of treelike structure. It dissolves in water to give viscous solutions which are relatively stable and which do not gel or retrograde under ordinary circumstances. Indeed, in solutions or pastes of the whole starch, the presence of the branched fraction has a moderating influence on retrogradation of the linear fraction, slowing down its precipitation and diminishing its gel tendencies. Nevertheless, the branched fraction does have outer branches of some 25 glucose units in length, still capable of some parallelwise association. The principal requirement is high concentration, so that these exterior branches are brought into close juxtaposition. While a 5% paste of waxy maize starch—all branched fraction—will remain clear and fluid for long periods of time, a 30% paste will rapidly harden to a gel. If we freeze and thaw a 5% waxy starch paste, the branched material will be transformed to an insoluble state, and this product will frequently give an x-ray diffraction pattern indicative of crystalline association of linear chains. The important difference between this sort of association and retrogradation of the linear fraction is the strength of the bonding forces. Retrograded linear material cannot be reversed even by autoclaving, while associated branched material can be readily restored to its original dissolved state merely by heating to 50 to 60°C. If we treat the branched material with beta-amylase enzyme, as shown in Fig. 4, the enzyme acts to strip off the outer branches, progressively splitting off maltose groups until its action is stopped when a point of branching is reached. The resulting "limit dextrin," with only short stub ends of the original exterior branches, shows no evidence whatsoever of retrogradation. It does not form a gel; it cannot be frozen out of solution. Similarly, there is the substance glycogen, occurring in animal livers and in certain shellfish. It is the counterpart of the branched starch fraction, except that the branch length is only 10 glucose units instead of 25 (Fig. 4). In other words, it is a bushlike molecule instead of a tree. No one has ever succeeded in making glycogen retrograde or gel or crystallize—its outer branches are just too short.

Now let's go back to the native starches, as they occur in the seed kernel of corn or in the tapioca root. We find that the starch substance is organized into minute spherocrystals or granules (Fig. 5), the size and shape of which are characteristic for the particular variety of starch, smallest in the case of rice starch, largest with potato. The individual granule of potato starch shows a series of concentric lamellations around the hilum, or botanical center of the granule, indicating the deposition of successive layers or growth rings (Fig. 6). When potato starch is examined under polarized light, the granules show the characteristic interference cross indicative of an orderly structure (Fig. 7). In addition to this layer structure, the granules appear to have a radial organization, something like a sycamore buttonball. For

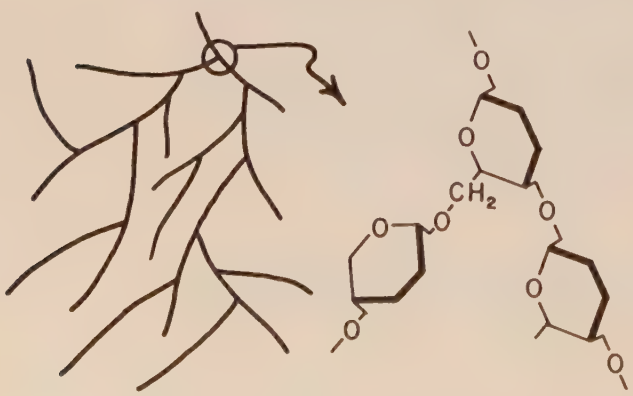

Fig. 3. Structure of the branched starch fraction showing configuration of the branch point

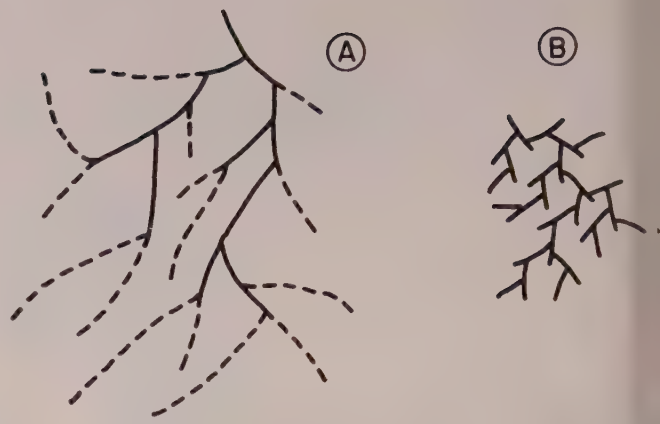

Fig. 4. A—Branched starch fraction, dash lines indicating exterior branches removed by beta-amylase; B—Glycogen molecule

when a dry granule is crushed under a microscope cover glass, it fractures into radial segments. The eminent Swiss polymer chemist, the late Kurt Meyer, has proposed the granule structure indicated schematically in Fig. 8. Here we have the randomly intermixed linear and branched fractions laid down with a radial orientation in a single layer of the granule. Wherever possible, the linear chains and linear segments of the branched fraction associate laterally to give parallelwise bundles which Meyer called "micelles." A long linear chain may pass through several of these micelles, or the outer fringes of branched molecules may participate in a number of such micelles. But between these organized areas are regions of looser and more amorphous character, where the chains and branches criss-cross in various degrees of randomness. When a granule is crushed, it may fracture through such points of weakness as those indicated by arrows in Fig. 8. This may necessitate the splitting of a primary valence bond by brute force, a concept which would be rank heresy in any other field but high polymers. But it requires a lot less energy to snap a primary valence bond than to disentangle a single molecule from its network of micelles. While the associative force between two hydroxyls on adjacent starch chains is relatively weak, remember that there are hundreds of such associative bonds.

The starch granule is insoluble in water because of this intricate interlacing of structure. Water may seep into the spongelike amorphous areas between the micelles, but this does not disturb the over-all crystallinity and intactness of the granule. When the starch granule is heated in water suspension beyond a certain critical temperature, the granules suddenly lose their polarization crosses and begin to swell. This transition or so-called gelatinization point occurs within a specific temperature range for each variety of starch, probably depending on the internal arrangement of starch substance within the granule and consequent variation in the imbibing of water. For example, potato starch granules lose their polarization crosses in the range of 56 to 67°C. when heated on a Kofler micro hot-stage, cornstarch in the range of 64 to 72°C. As the granule is progressively heated in water above the gelatinization temperature, it continues to swell until its outlines become vague under the microscope. Nevertheless, it can be shown that the swollen granule still persists as an elastic entity, even in well-cooked pastes. Indeed, the viscous consistency of a cooked starch paste is due primarily to these large swollen granules, jostling one another and stringing out when the paste is stirred. Prolonged cooking or vigorous stirring may fragment many of the swollen granules, thus reducing the viscosity of the paste, but the major portion survives as intact entities. Only by autoclaving can the swollen granule be dissolved.

There is a simple and useful way of following the gelatinization of starch, by graphing the changes in viscosity when a

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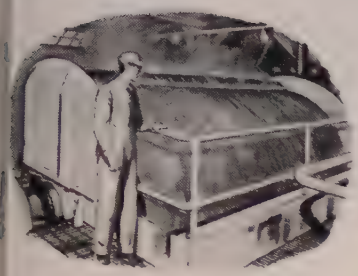

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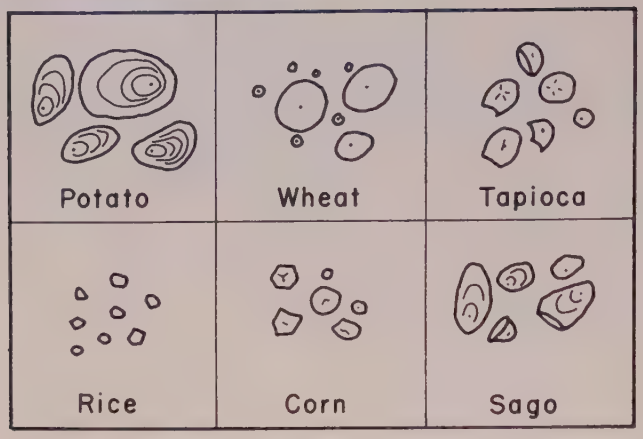

Fig. 5. Microscopic appearance of various starch granules

suspension of starch granules in water is heated at a uniformly rising rate. This is most conveniently accomplished by means of the Brabender Amylograph or the Bechtel viscometer, which plots increase in viscosity over the gelatinization and swelling range. In Fig. 9 will be seen the pasting curves of several typical starches. Tapioca gelatinizes with a peculiar and unexplained double "hump," then thins down as the swollen granules are distorted and fragmented by cooking and by mechanical agitation. In comparison, cornstarch gelatinizes at a substantially higher temperature, does not give the high peak viscosity of tapioca, but likewise does not show the drastic thinning on continued heating.

Meyer's concept of the micellar organization within the granule provides a very adequate explanation of the process of gelatinization. Prior to the gelatinization point, water merely soaks into the open intermicellar areas. When the gelatinization temperature is reached, the loose associative bonding in these areas is rapidly satisfied by hydration, and the granule begins to swell tangentially. Swelling continues as the temperature is raised and more water seeps into the intermicellar areas. Some of the shorter linear chains may be released from their entanglement and leach out into the surrounding substrate. But most of the branched molecules are too deeply enmeshed to follow suit. Through all this swelling, the crystalline micelles remain intact, and so eventually we arrive at an enormously swollen elastic network of molecules tied together at frequent points by the persistence of the micelle (Fig. 10). If this paste is violently agitated, as in the Waring Blendor or by ultrasonic radiation, the terrific opposing stresses may shear glucosidic bonds, rather than

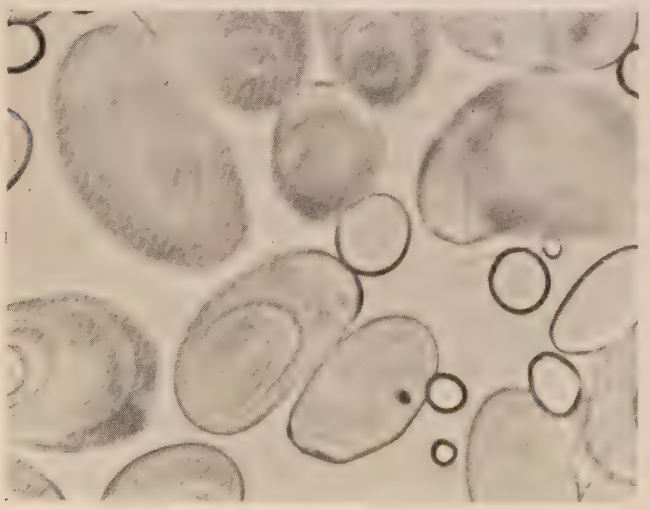

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Fig. 6. Photomicrograph of potato starch granules

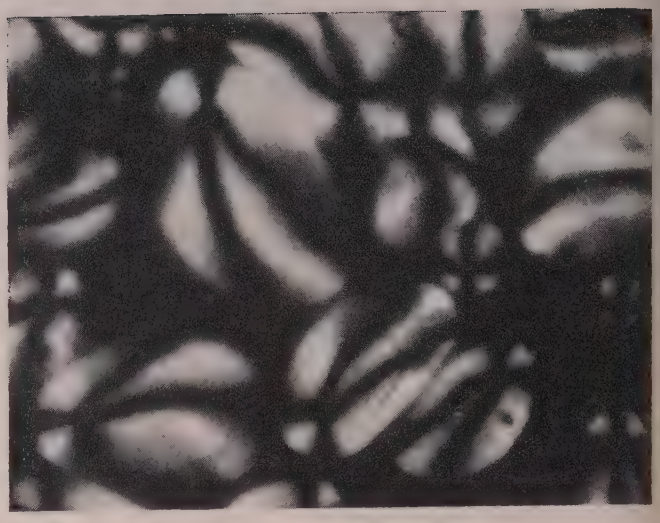

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Fig. 7. Potato starch granules under polarized light, same field as Fig. 6

tear the individual micelle apart. The granule owes its existence and swelling properties to these micellar areas, and not specifically to the presence of either fraction, since both waxy starches and high-linear wrinkled pea starch form optically anisotropic granules. However, the content of linear material does have a substantial influence on gelatinization temperature and on swelling behavior of the granule. Waxy maize starch gelatinizes some 6 to 7°C. lower than ordinary cornstarch, while wrinkled pea starch gelatinizes with difficulty at much higher temperature and does not disperse or dissolve even on autoclaving. So it would seem that the higher the linear content, the stronger is the intermolecular association within the granule.

There is one further phase of fundamental starch chemistry which must be mentioned before we get down to practicalities. This concerns the interaction of the linear fraction with certain polar agents such as the various higher alcohols, fatty acids, and sulphonated oils, where we have a hydrophilic hydroxyl, carboxyl, or sulphonic group attached to a relatively large alkyl or aryl residue. These agents have a special affinity for the linear fraction of starch, forming an insoluble complex which precipitates from solution. While the fractionation of starch is not within the province of this paper, it might be mentioned parenthetically that the first satisfactory separation of the starch fractions was accomplished in our laboratories by selective precipitation of the linear component with butyl or amyl alcohol. The development of this technique has been of very substantial assistance in formulating our present concepts of starch.

If we take a starch paste, autoclave it to effect dispersion and solution of the swollen granule, then add a sulphonated

Fig. 8. Micellar association within the unswollen starch granule, according to K. H. Meyer. Micelles indicated by thickened sections due to lateral association of chains. Arrows indicate fracture points

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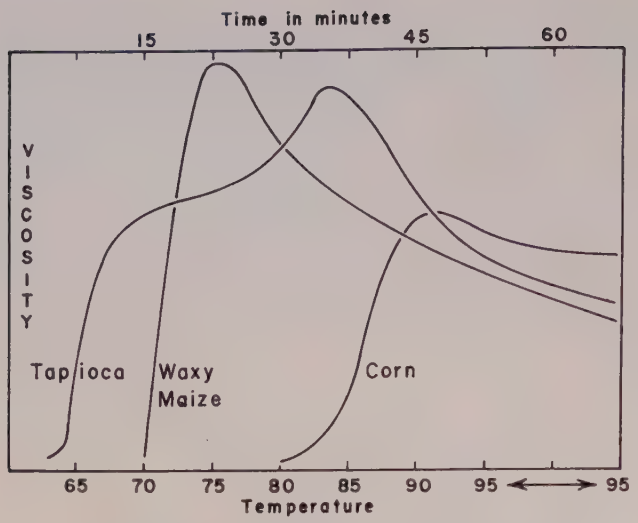


Fig. 9. Gelatinization curves of various starches, as determined with the Brabender Amylograph

oil or a higher alcohol, such as cyclohexanol, the linear fraction is quantitatively precipitated as an insoluble complex, leaving the branched fraction in solution. This mixture can be boiled without redissolving the insoluble complex. If we heat an aqueous suspension of granular starch in the presence of cyclohexanol or sulphonated oil, we find that gelatinization and swelling of the starch are markedly retarded. The granule undergoes only a restricted swelling, and does not fragment or dissolve on continued cooking. Indeed, if we autoclave such a paste at 20 lb. pressure, then pass it hot through a Sharples supercentrifuge, we find the total starch substance deposited in the centrifuge bowl with no starch solubles in the supernate. If we now repeat these same operations on waxy maize starch, containing no linear fraction, we find that the polar agent has no effect whatsoever on the behavior of the waxy granules. They gelatinize at the same temperature as in water, swell freely, and dissolve on autoclaving. Hence we attribute the effect of polar agents on the normal starches to the formation of an insoluble complex with the linear fraction, waterproofing the granule in the sense that the imbibing of water and solution of the granule are markedly reduced. This same effect shows up in other ways if tapioca or potato starch is cooked in the presence of sulphonated castor oil. Instead of the normal clear, slimy pastes, we obtain

Fig. 10. Micellar association within the swollen granule, according to K. H. Meyer

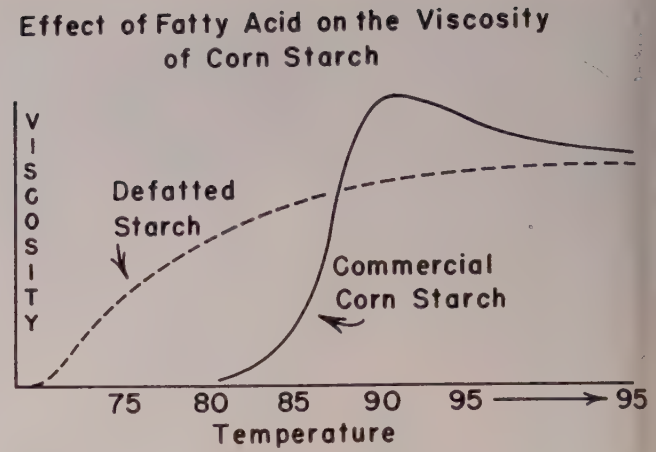


Fig. 11. Gelatinization curves of commercial and defatted cornstarch, showing the influence of fatty acid

opaque pastes with a "short" thick consistency. The swollen granule is toughened, loses its elastic qualities and its ability to string out.

Cornstarch contains a small amount, 0.6%, of associated fatty acid, tied up as an insoluble complex with the linear fraction. And even this small amount of polar material alters the behavior of the starch, toughening the granule against gelatinization and swelling. We can remove this fatty acid by exhaustive extraction with ethyl alcohol. If we then gelatinize this defatted starch in the Brabender recording viscometer, we obtain a curve shown in Fig. 11. If we add a small amount of fatty acid or sulphonated castor oil to the defatted starch, the gelatinization curve is restored to that of the original nondefatted starch. Indeed, with excess amounts of these agents, we can displace the curve even further to the right.

I have said that the linear fraction of cornstarch is of optimal chain length to give rigid elastic gels. Defatted cornstarch gives even stronger gels than ordinary commercial cornstarch, because a portion of the linear fraction in the latter is tied up as an insoluble complex with the natural fatty acids. And it is primarily the linear fraction which is responsible for gel formation. The addition of sulphonated oil is frequently used in the textile industry to prevent formation of gels in the size kettle. But it seems preferable to add the sulphonated oil *after* the starch has been thoroughly cooked, thereby precipitating the linear fraction without impairing gelatinization of the granule and consequently immobilizing the

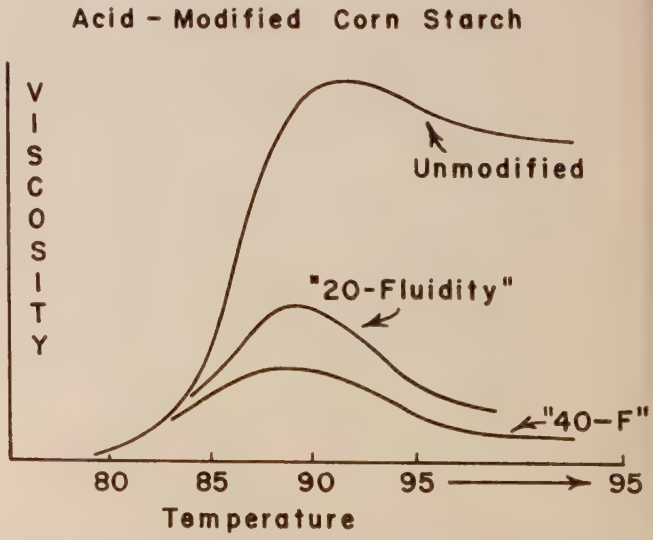
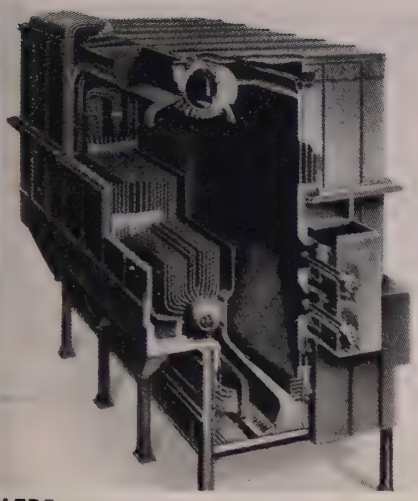


Fig. 12. Gelatinization curves of thick-boiling and two thin-boiling cornstarches



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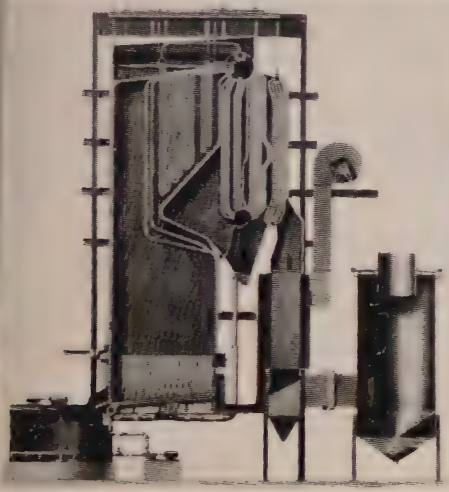

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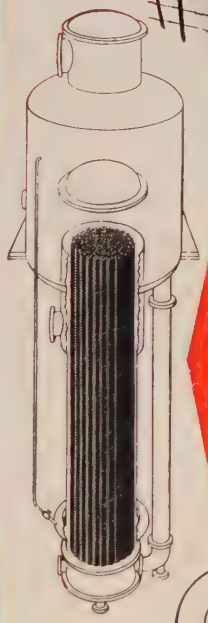

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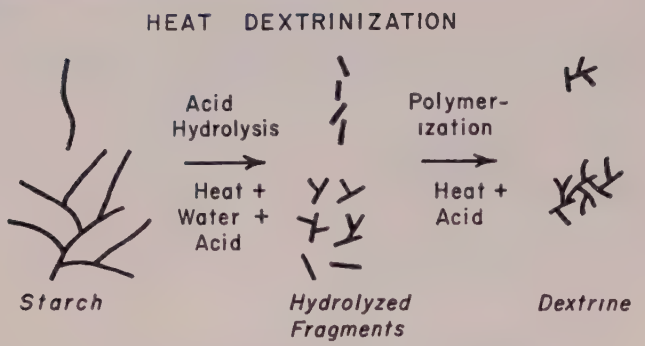

Fig. 13. Mechanism of hydrolysis and repolymerization of the starch fractions during dextrinization

branched fraction. These considerations would likewise seem to apply to the paper coating field, since sulphonated castor oil is a frequent adjunct in your formulations.

The starch granule was designed by Nature as reserve food supply for the growing plant, not as a paper or textile size. Hence its characteristics must be modified for most uses. Essentially, there are only two reasons for modification—first, to reduce viscosity to more suitable levels for certain specific applications, and second, to reduce the various retrogradation effects of the linear fraction. Too frequently, I believe we accomplish one of these purposes somewhat to the detriment of the other.

Starch has five main fields of commercial application:

1. As a gel former of which the old-fashioned cornstarch pudding is an excellent example.
2. As a thickening agent for such diverse purposes as gravies and electrical dry cells.
3. As an emulsifier or protective colloid, as in salad dressings and oil-well drilling muds.
4. To provide a smooth flexible adherent coating.
5. As an adhesive.

Of these five applications, you are vitally interested in the last four, and you have sometimes been plagued with the first.

Let us take a look at commercial methods of starch modification—both present and prospective—to see what happens fundamentally to the starch and how this influences its use. One of the commonest modified starches is the thin-boiling type, prepared by suspending granular starch in warm dilute acid at a temperature just below the gelatinization range. Under these circumstances, the acid soaks into the more open intermicellar areas, hydrolyzing a few glucosidic bonds here and there without affecting the granule form. However, when such a starch is subsequently gelatinized in hot water, the connecting network between the micelles has been weakened, and the granules fall apart to give pastes of reduced viscosity. The pasting curves of several such starches are shown in Fig. 12.

The action of starch-splitting enzymes is much the same as acid. The enzyme of saliva is one of the few which acts on the ungelatinized starch granule, seeping into the intermicellar areas and saccharifying the starch substance in these areas, but leaving the micelles intact. The end product of this reaction (termed "amylodextrin") therefore consists almost entirely of the short linear chains which comprised the original micelles. It shows the anticipated properties, dissolving in hot water and crystallizing on cooling. The ordinary commercial liquefying enzymes only act on the starch granule when the intermicellar areas are opened up somewhat by moderate swelling. But here again, the primary thinning action is due to hydrolysis within the intermicellar network, without much attack on the resistant micelle.

In the manufacture of thin-boiling starches by the wet acid process, we can only go to a certain thinness without incurring the formation of excessive solubles. Since the process is conducted in aqueous suspension and the starch subsequently neutralized, filtered, and washed, these soluble would represent a serious yield loss. So the higher modifica tions of thin-boiling starches are prepared by heating dry starch in a dextrin cooker with traces of hydrolyzing acid These products—known as white dextrins—are used as adhe sives and sizing agents.

These three types—thin-boiling starches, enzyme-liquefied starches, and white dextrins—all represent a simple hydrolytic conversion, primarily in the intermicellar network. In no case is the linear fraction destroyed; it is merely broken down to shorter fragments. Consequently, these products exhibit the specific retrogradation behavior peculiar to their chain length. If relatively long, they form gels; if shorter, they give precipitates.

Quite frequently, this precipitation of short linear molecules would be definitely detrimental for a specific use, as for example, in the manufacture of a gummed paper tape where a clear stable adhesive is required to coat the paper base. A fourth type of modified starch—comprising the various gums and yellow dextrins—is suited to this use. They are likewise manufactured by heating dry starch with a trace of acid, but for a longer time and to a higher temperature than with the white dextrins. As shown in Fig. 13, the starch substance first undergoes a preliminary hydrolysis to small fragments corresponding to the white dextrins. As the temperature is raised and moisture eliminated, these fragments appear to recombine to give some sort of branched type of structure. The viscosity of the product may even rise during this repolymerization, indicating an appreciable increase in molecular size. While the precise mechanism of repolymerization is not known, the net effect is to reduce the content and influence of the linear fraction. The manufacturer of gummed tape can apply a solution of such a yellow dextrin without the dextrin thickening up or becoming opaque during application. Moreover the dried film remains transparent and glossy and retains the ability to remoisten rapidly and readily.

However, in each of these various modifications, we have drastically reduced the viscosity of the starch substance since the branched fraction is hydrolyzed or dextrinized right along with the linear fraction. And viscosity is a most useful property. Where maximum colloidal qualities are desired, it would seem more appropriate to alter the linearity of the linear fraction, rather than to smash the whole starch to bits. This would reduce retrogradation effects without sacrificing too much of the useful colloidal properties of large molecules. One way of accomplishing this is by oxidation. When granular cornstarch is treated with a solution of alkaline

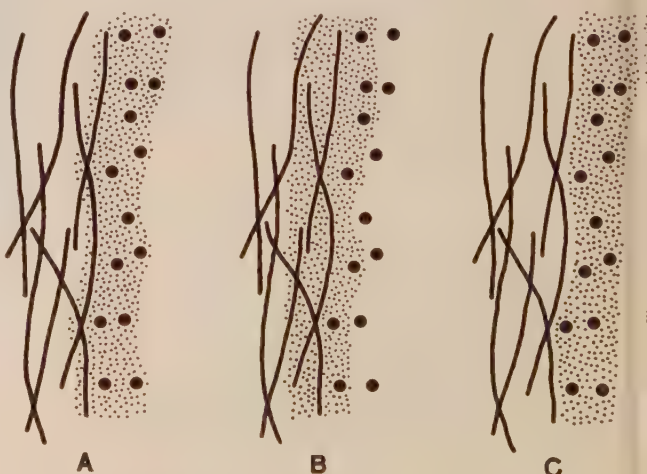

Fig. 14. Schematic representation of the penetration of clay coating into fibers of the sheet. Black circles represent clay particles, shaded area represents the starch substrate. Diagrams A, B, and C indicate proper, excessive, and insufficient penetration, respectively

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hypochlorite, various glucose units are oxidized at random, sometimes with the introduction of carbonyl groups on carbon 6, sometimes at carbons 2 or 3. While both linear and branched fractions are oxidized, the technological benefit lies in oxidation of the linear molecules. Seemingly, if a linear molecule is oxidized at two or three points along its length, it becomes warped or distorted by these discontinuities so that it will no longer associate or retrograde. Such modified starches give relatively clear solutions and provide excellent protective colloid action.

Unfortunately, hypochlorite oxidation likewise causes some sort of obscure scission of starch molecules, to give products of reduced viscosity. Much the same reduction of linear character can be accomplished by a slight etherification of the starch, without attendant loss of viscosity. For example, an alkaline starch suspension may be treated with ethylene oxide to introduce hydroxyethyl groups into the starch. While both fractions are affected, here again the major benefit is in derivatizing the linear fraction. A very minor proportion of derivative groups, perhaps one on every 10th or 20th glucose unit, is sufficient to produce obstructions on the linear chain which interfere with side-by-side association. Snagging the zipper action, as it were. Hence these starches give relatively clear and stable pastes which show a minimum of gelation and skinning. Moreover, any desired viscosity can be achieved by derivatizing the appropriate thick or thin-boiling starch. The only trouble is that the cost of derivatizing agents makes these products relatively high priced.

While my personal knowledge of the clay-coating field is somewhat limited, your basic requirements seem to fit into the same pattern as the use of starch in any other type of coating. So I am going to describe what I think our starches are doing in your clay coatings, and try to visualize the optimal product for the purpose.

First of all, you want a certain viscosity. I picture the cross section of a sheet of coated paper somewhat as depicted in Fig. 14, a mat of cellulose fibers of somewhat open structure. The coating adhesive should be of such a viscosity that it will penetrate slightly into this surface, but only enough to surround the surface fibers. If the starch medium is too thin (as in the second diagram), then it filters off into the paper, leaving the clay particles high and dry, with nothing to attach them to the sheet. If the starch solution is too thick (as in the third diagram), it will not penetrate around the surface fibers and hence the coating will not be properly interlocked with the paper. So viscosity is a primary consideration.

But we can attain the same viscosity either by use of a high concentration of a thin dextrin, or by use of a lower concentration of a thicker starch product. What is the fundamental difference? I don't think it is basically a matter of adhesion of starch for clay or starch for cellulose fibers, though obviously the starch must wet and surround these surfaces. Instead, the strength of a coating would seem to be an internal affair, the cohesion of starch to starch. For a moment, let's leave retrogradation out of the picture. Let us consider the analogy of a bush, progressively chopped into smaller and smaller fragments. If at various stages in this disintegration, we throw the pieces together as a sort of pile of jackstraws, where do we get the greatest intermeshing and interlocking? I think it will be at an intermediate stage, when the bushlike structure is broken down sufficiently to compact fairly well but where the branching twigs still extend to some extent through the brush pile. It would be difficult to pull apart such a mass, and practically impossible to separate one twig without disturbing the others. But if we further disintegrate our bush down to matchstick dimensions, then it has no internal strength at all. We can pull out fragments either singly or by the handful without disrupting the pile. So in a clay coating, the cementing starch must have a certain internal strength, a continuity of enmeshment around the clay particles and the surface fibers. Any forcible break is most likely to occur within this matrix of starch molecules, and not at the interface between starch and clay or starch and cellulose.

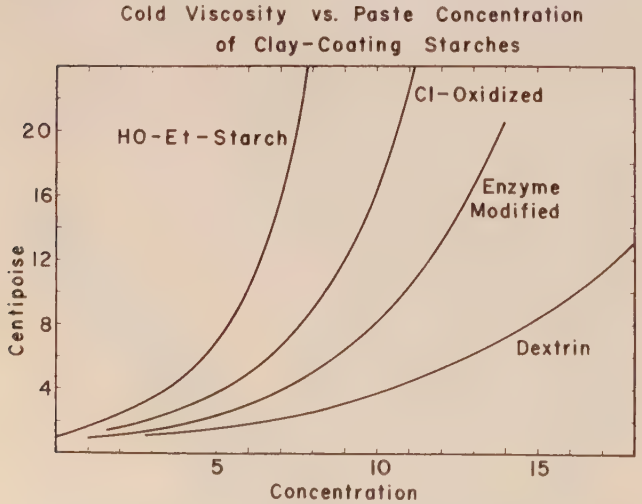

Fig. 15 Comparative viscosities of various clay-coating starches as a function of concentration

In modifying starches for clay coating, I believe we have traditionally gone too far to the thin side, fragmenting the molecule to the point where it loses much of its internal strength. We have had to do this to avoid the ill effects of retrogradation. You are coating with relatively cold pastes, necessarily using the least possible amount of water. And retrogradation is most pronounced at low temperatures and high starch concentrations. Any slight association to a gel structure will give coatings which do not level off properly, showing a tendency to "feather" at the nip of the rolls. Yet if the starch is fragmented to the point where it shows no gel tendency, then it may begin to crystallize as discrete particles which have no usefulness whatsoever.

So you have had to sacrifice some internal molecular strength for absence of retrogradation. If the unpleasant associative effects of the starch could be reduced by oxidation or by derivatization, then it should be possible to use a clay-coating adhesive of larger molecular size. This would provide not only greater internal strength but likewise better protective colloid action. You are now using products of widely different molecular size, as indicated by the viscosity-concentration curves shown in Fig. 15. Here we have three starch products traditionally used in clay coating: a cornstarch dextrin, an enzyme-modified starch converted to about the level which you employ, and a conventional chlorine-oxidized starch. Also included is a thin-boiling hydroxyethyl starch which I understand has been used at least experimentally in coatings. Since these viscosity curves are intimately related to molecular size, it will be appreciated that the dextrin and enzyme-modified starch are fragmented to much smaller molecular dimensions than the hydroxyethyl and oxidized starches. Hence I feel that the latter starches will yield stronger coatings than the dextrins and enzyme-modified products.

As qualitative evidence for this theory, may I mention a simple technique which we have been trying to develop as a criterion of starches for textile sizing. Our argument is this—that any starch which is going to give a strong continuous film on the surface of a cotton fiber should likewise yield a strong coherent film when coated out and dried on a glass plate. Figure 16 shows the sort of films obtained with the same four clay-coating adhesives mentioned above. Films of the dextrin and the enzyme-modified starch have no internal strength—they crack and splinter into fragile flakes. In other words, the molecules have been reduced to matchstick dimensions. The third and fourth films are chlorine-oxidized and hydroxyethylated starches, respectively. These products

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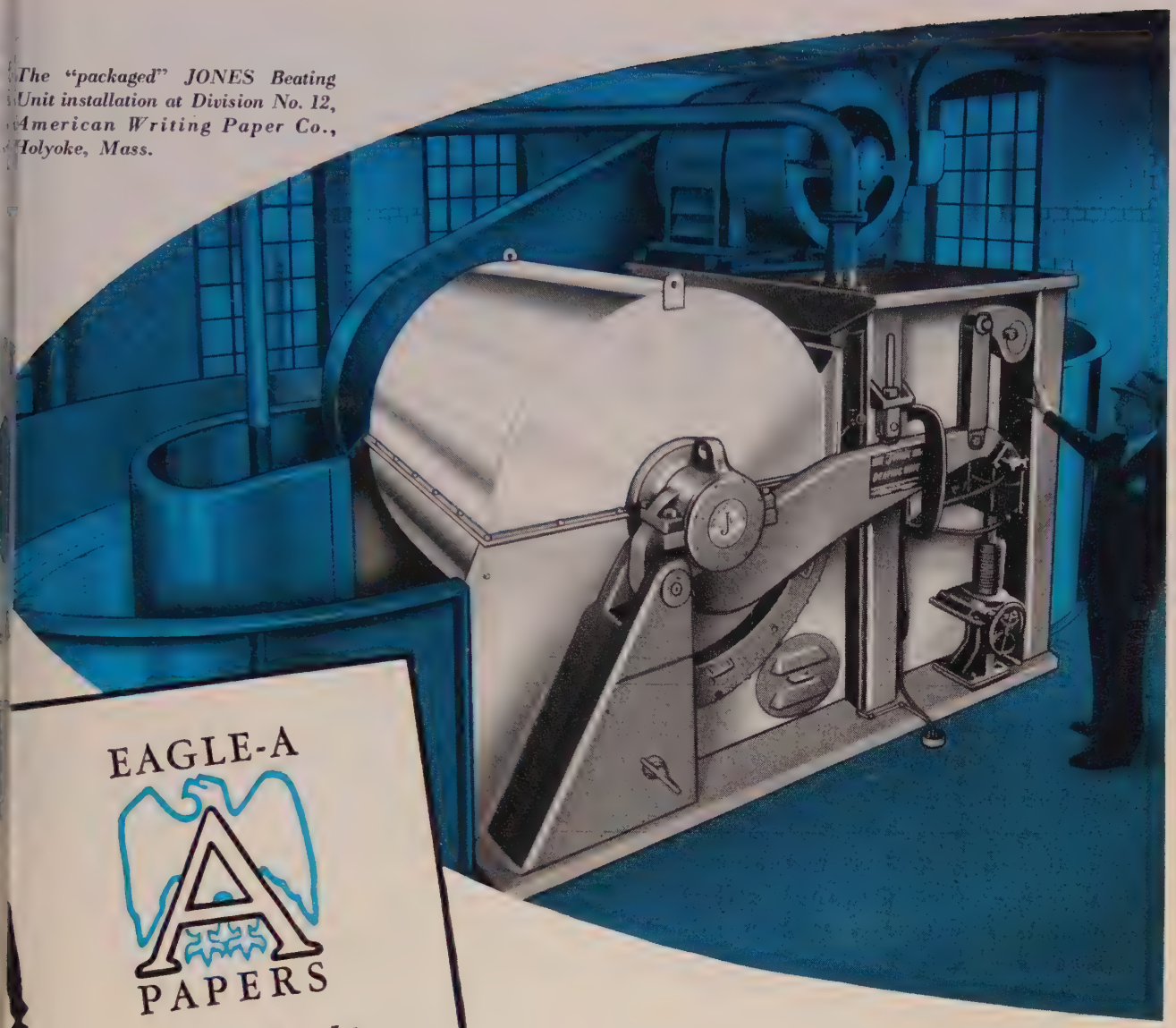

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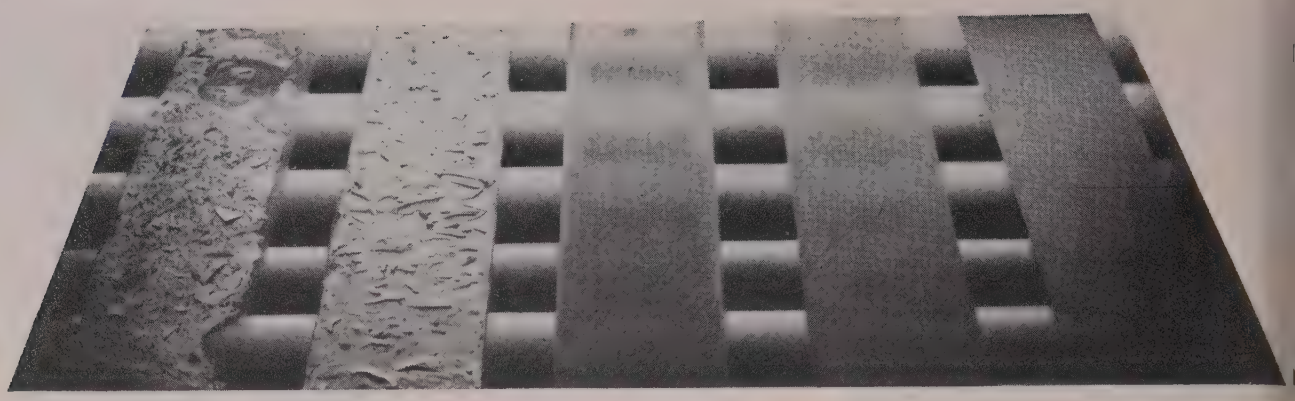


Fig. 16. Simple films of various modified starches, coated on glass, respectively dextrin, enzyme-modified cornstarc chlorine-oxidized starch, hydroxyethyl starch, and 75-fluidity thin-boiling starch

yield continuous films which adhere to the glass with a minimum of cracking. They can even be scratched with a knife point without excessive chipping. The fifth film is a 75-fluidity thin-boiling starch, much too thick for your purposes, but merely showing that the starch molecules do have film strength if they are not excessively fragmented. However, such a product shows too great a tendency to gel; it could not be used unless it were applied hot enough to delay gelation until the coating had leveled off. Indeed, this particular film had to be coated hot, and even under these circumstances it gave a somewhat rough, dull surface.

Mr. Frost has asked for some sort of criterion of the effectiveness of starch as a coating material. The actual casting of a simple film may have usefulness in this connection. There is another test which we have been investigating. This involves measurement of the turbidity of dilute starch pastes and dextrin solutions, as a relative index of their tendency to retrograde. In Fig. 17, we have clarity versus concentration curves for the same products whose film-forming qualities were described above. In general, a starch product which yields a clear, stable solution should likewise give a continuous film of good strength. Opacity in the starch paste would represent precipitation of linear aggregates; if this occurred in the coated film, it would disturb its continuity and weaken its strength. In this same connection sulphonated oils do not actually plasticize the coating; they merely precipitate linear material which may be causing gelation trouble. Similarly, soap may be added to prevent "feathering" on the rolls, another fault which can be attributed to gel tendency of the linear fraction. In neither case does the precipitated linear fraction contribute any strength to the coating.

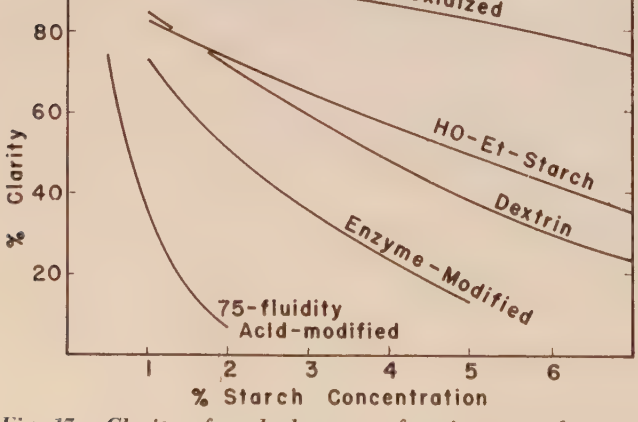


Fig. 17. Clarity of cooked pastes of various starch types as a function of concentration

An ideal starch coating is not merely a brush pile of lo jackstraws. Were this the case, the dried coating would loose and porous, of low density and poor brilliance. would penetrate too deeply. Also, such a coating would easily permeated and even redissolved by water—there wo be nothing to anchor the starch molecules. Associative bo ing between molecules probably does and should come i play to a considerable extent after the coating is applied as the water is dried off. This would have the effect of co pacting and tightening the network of molecules around clay particles and surface fibers, thus giving a denser, smooth more resistant finish. While not actually impervious water, such a network might be considered as partially cu by cross-bonding between molecules, at least to the ext where water does not freely dissociate and dissolve the mo cules. The important point is that this associative bond should take place *after* deposition of the coating. The con tions of high-speed coating tend to maintain the molecules a dissociated state until they have been deposited. This merely the phenomenon of thixotropy, the momentary sociation of intermolecular forces under high shear. A the coating is laid down, the quiescent molecules have leisure to orient themselves, to zipper together as the coat dries. You cannot use heat to dissociate the starch, as textile mills do, but you can use shear.

So I believe optimum results will eventually be reali by the proper combination of two different starch mod cations:

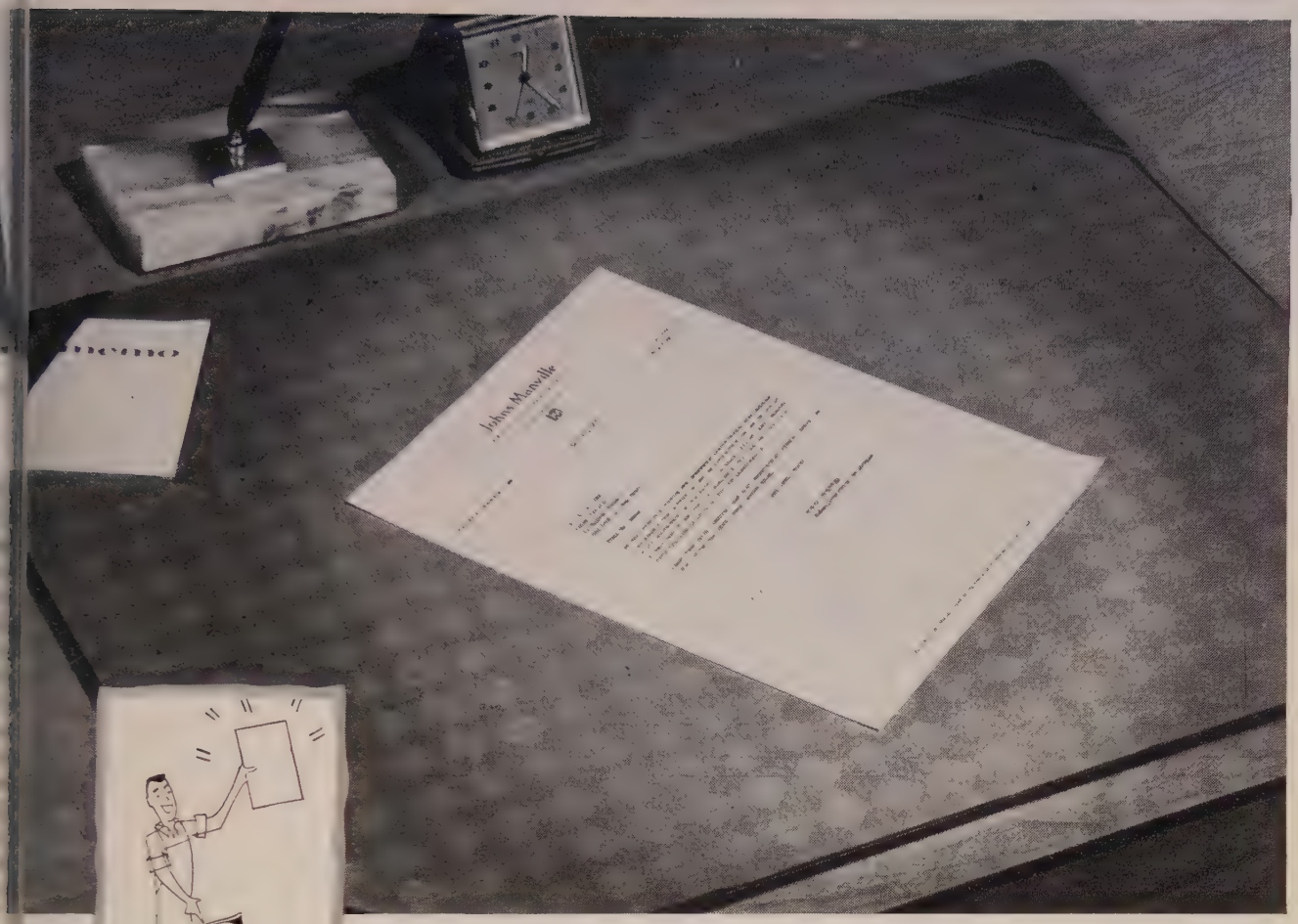
1. Fragmentation of the starch molecule only far enough produce the required viscosity at the desired starch c centration.
2. Warping the linearity of the starch substance j sufficiently to overcome the unpleasant effects of re gradation without sacrificing the advantages of some association in the finished coating.

The starch industry has already laid the groundwork such a product by several alternative approaches: the co mercial development of waxy starches, investigation of bet oxidation methods, and the growing field of starch esters a ethers. I believe the basic principles have already been covered—we now need the tedious process development w to establish the proper balance of qualities, to find out j how much we have to twist the arm of the starch molec to make it behave.

I have already answered some of Mr. Frost's questio Suppose we check through his list:

First: Water retention. This seems to require that starch molecules be as large as possible, consistent with ot requirements. Big molecules have good water-hold capacity, preventing the water from draining off into the sto Small molecules have much less ability to bind water.

Second: Penetration of size into body stock. Here aga



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the molecules should be as large as possible. Big molecules have a blocking effect, preventing the adhesive from penetrating too far into the pores of the body stock. Actually, this is merely another way of saying that big molecules have an inherent viscosity. On the other hand, we do not want the sort of viscosity or pore-blocking which would be caused by the presence of swollen granules or fragments of granules.

Third: Adhesive strength of starch versus casein. This one I can't answer, since I do not have a speaking acquaintance with the casein molecule. I can only venture to hope that we can get an equivalent product (certainly costwise) by proper manipulation of the starch molecule.

Fourth: Leveling of the wet coating. I think this can be blamed entirely on gelation of the linear fraction. It should be minimized either by use of the waxy starches or by modes of modification which disturb the linearity of the starch without too much fragmentation.

Fifth: The fundamental reasons for starch viscosity. This subject has been adequately treated. Viscosity is entirely explicable on the basis of the size of molecules and the associative network between them.

Sixth: "The Case of the Undisorganized Starch Granule." I have examined the starch sample which Mr. Frost has discussed. The trouble is due to a small amount of aggregated starch granules, groups of ten to a hundred granules stuck together by partial gelatinization. Consequently, these aggregates persist as "microlumps" through the cooking operation and appear as specks on the final finish. This so-called "residue starch" represents improper processing, and fortunately it doesn't occur very often. The cause is difficult to determine with certainty, and my best guess is faulty drier operation, tiny droplets of condensate water contacting hot starch. The problem is definitely in the lap of the engineering department of the starch processor.

Seventh: Dusting on the calender rolls. I would say that the starch molecules have been excessively fragmented, so that the coating does not have the internal tenacity to resist the punishment of the calendering operation.

Eight: Folding without cracking. This seems to require a certain amount of plastic flow within the coating, a slippage between starch molecules. A starch-coated sheet can be folded without cracking if it is conditioned at high humidity. Water acts as a plasticizer, simply meaning that some of the associative bonding of the starch is satisfied by adsorption of water, thus reducing the starch-to-starch linkage. Perhaps suitable plasticizers can be found. Or perhaps intermolecular association can be reduced by a higher degree of etherification or oxidation. But in any case, my guess is that foldability can be realized only at the expense of strength of the coating, as reflected in a lower wax pick test.

Ninth: New developments in enzyme chemistry. There has been substantial fundamental progress in enzyme chemistry in the past several years. A number of the amylases have been isolated in pure crystalline form. An enzyme has been discovered in potato juice which is capable of making or breaking the branching point in starch. Another enzyme has been tentatively reported which transforms linear starch substance into branched molecules, apparently doing a much better job than we can ever hope to do by dextrinization. But my guess is that these enzymes are going to be laboratory curiosities for a long time to come. I prefer to put my faith in modification by chemical means.

Tenth: A waterproof cure of starch on the alkaline side. Suppose I pass this one, merely saying that there is substantial reason to hope for a copolymerization of starch with a cross-bonding agent at high pH levels.

It is only by a full mutual understanding of these factors that we can hope to find an eventual solution of the problem. We are therefore indebted to Mr. Frost for delineating the problem, and I can assure you that the starch industry is endeavoring to meet your needs.

Modifying the Flow Properties of Starch-Pigment Coatings

W. C. GALLAGHER

THIS paper is concerned with the factors which influence the flow properties of high-solids pigment coatings in which starch is used as the adhesive.

To begin with, an enumeration of the major factors which affect the flow properties of coating mixtures should be presented. These are: type of pigment, type and amount of adhesive, type and amount of deflocculating agent, presence of additives, machine speed, and temperature of the coating mixture.

The pigment makes up the principal part of the coating solids and exerts an important influence on the flow properties and also on the pattern, brightness, gloss, and other important physical properties of the coated paper. A sizable effort has been expended in studying the effect of pigments in coating. The fascinating research which has been done has culminated in the development of the modern coating pigments for use in high-solids coatings. This work has been fully covered in the literature and no attempt was made to study pigments in this program.

Early in the application of starch in coating it was found that the type of modification as well as the extent of modification of the starch used had a pronounced effect on the flow properties of the prepared coating. It was found that coatings with substantially different flow characteristics were obtained with the different types of modified starches. The effect of degree of modification was more intensively brought home to the coating formulator when the practice was begun of converting native starches in the mill with enzymes. Today enzyme-converted, oxidized, and dextrinized starches are all widely used as coating adhesives in a variety of applications involving both low and high solids coatings. Each type of starch has its own particular advantages.

Soaps of various kinds have been part of coating formulas almost from the beginning of modern coating practice. Originally soap was added to minimize dusting during supercalendering, but more important is the effect which soap has on the plastic viscosity and leveling properties of high-solids coating mixtures.

The effect of deflocculating agents on clay and other pigments has been investigated in great detail by a number of workers. As a result of this work it is possible to choose the proper kind and amount of deflocculent for any pigment mixture. The importance of deflocculating agents to the flow properties of the coating mixture is well recognized.

DISCUSSION

In the interest of simplifying the work, the program has been limited to the study of a few variables. The basic formulas have been reduced to essentials so that each factor investigated can be observed with as little complication as possible. In the basic coating formula a slurry of high grade domestic clay containing 70% solids was dispersed with a fixed amount of deflocculating agent. To this were added the other materials under study. All measurements of flow properties of the coating mixtures were made at a temperature of 80°F., unless otherwise stated.

The three principal variables studied were:

1. Type of starch. The type of starch has been designated as the variable factor in a series of experiments designed to show the variation in flow properties which can be brought about by changes in adhesive composition. Values illustrating the effect of time of standing on the flow of coating mixtures are also included in the discussion.

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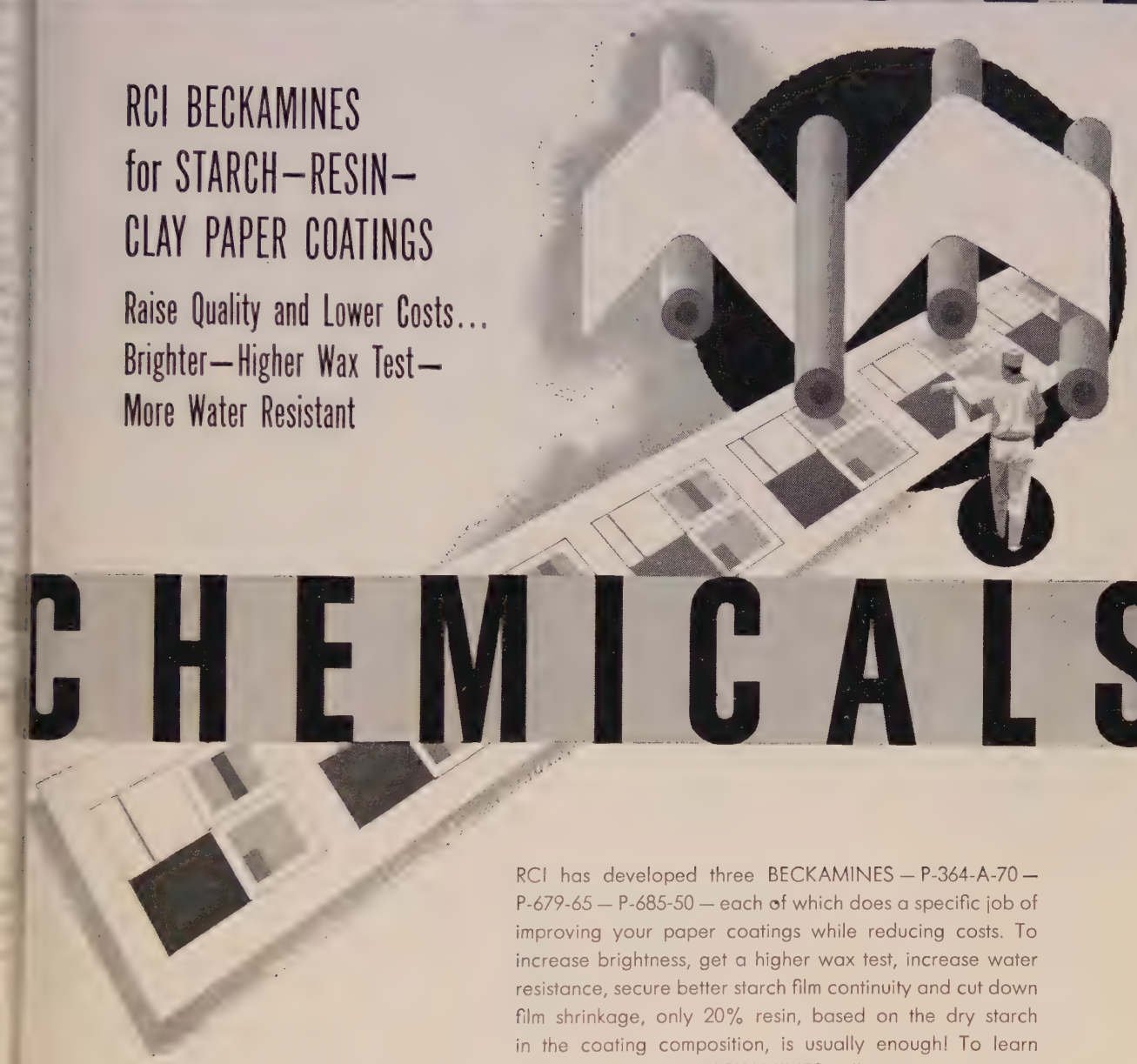

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Table I. Effect of Type of Starch on Flow Properties of Coating Mixtures

5% Soap on adhesive

Adhesive	Brookfield viscosity, cp. Fresh	Brookfield viscosity, cp. Aged 24 hr.	Plastic viscosity 500 r.p.m.	Plastic viscosity 1000 r.p.m.	Leveling index
100% Special coating dextrin	1,340 (20 r.p.m.)	1,300	1.80	1.49	0.30
70% Special coating dextrin, 30% oxidized starch	2,860 (20 r.p.m.)	4,400	2.41	1.94	0.35
50% Special coating dextrin, 50% oxidized starch	7,700 (20 r.p.m.)	13,000	3.21	2.48	0.43
100% Oxidized starch	32,480[a] (10 r.p.m.)	126,000[a]	3.86	2.90	0.48
100% White dextrin	3,100 (20 r.p.m.)	5,740	1.72	1.44	0.30
100% Enzyme-converted starch	11,200 (20 r.p.m.)	.....	3.05	2.39	0.40

[a] Different spindle.

2. Type and amount of soap. The effect of varying the type and amount of soap has been used as the principal variable in a number of experiments.

3. Effect of stabilizing agents. Frequently there is some delay between the preparation of the starch solution and the time of its incorporation into the pigment slip. During this interval retrogradation of the starch may occur, particularly with enzyme-converted starches, causing an undesirable increase in the viscosity of the solution. The effect of "liquefying" agents to combat this phenomenon and the stabilization which may be gained are briefly studied in one set of experiments.

Until recently research workers did not have a tool with which to measure the effect of the severe stresses to which a coating is subjected on the modern high-speed coater. The development of the Hercules Hi-Shear viscometer by Smith and his associates (*1*) has provided such an instrument and our knowledge of the effect of high shearing stresses has been considerably expanded through the use of this instrument. These values are useful in illustrating the increase in viscosity which takes place on standing.

RESULTS

It must be kept in mind in interpreting the following results that the particular values obtained apply only for the set of conditions used. Different results would be expected if the pigment composition, solids content of the coating mixture, or other conditions were different from those used.

In the following study, oxidized starch, a special dextrin, enzyme-converted starch, and blends of dextrin and oxidized starch were used. The results obtained have been presented in table form.

Effect of Type of Starch

Table I contains rheological information on coatings made

Table II. Effect of Type of Starch on Flow Properties of Coating Mixtures

3% Soap on Adhesive

Adhesive	Brookfield viscosity, cp. Fresh	Brookfield viscosity, cp. Aged 24 hr.	Plastic viscosity 500 r.p.m.	Plastic viscosity 1000 r.p.m.	Leveling index
100% Special coating dextrin	1,000 (20 r.p.m.)	1,400	1.44	1.29	0.17
70% Special coating dextrin, 30% oxidized starch	2,400 (20 r.p.m.)	4,100	2.05	1.72	0.28
50% Special coating dextrin, 50% oxidized starch	8,200 (20 r.p.m.)	60,000	3.02	2.26	0.47
100% Oxidized starch	146,000[a] (4 r.p.m.)	232,000[a]	..	..	..
100% White dextrin	5,400 (20 r.p.m.)	29,000	2.01	1.62	0.34
100% Enzyme-converted starch	7,600 (20 r.p.m.)	..	2.89	2.22	0.44

[a] Different spindle.

While this instrument is not universally accepted as yet, it appears to offer the first scientific approach to the subject of high shear rheology, and several coating mills have found it of great value for coordinating laboratory evaluations with mill results.

In applying the instrument to this study certain concepts which have been laid down by the designers have been followed (*2*). A value designated as the "leveling index" is used as an indication of the pattern-forming characteristics of a coating color. This index has been defined as the ratio of the coefficient of thixotropic breakdown to the plastic viscosity. In this study the commonly used minimum value of 0.3 is taken to be the lowest value at which a coating can be expected to apply without producing an undesirable pattern.

For measuring the viscosity at low rates of shear, a Brookfield viscometer has been used. Values were obtained on both the fresh coating mixtures and after standing for 1 day.

with various combinations of a special coating dextrin and oxidized starch, both of low viscosity. Two other products are also evaluated. These coatings were made with the clay slurry described above, using 0.3% tetrasodium pyrophosphate on the weight of clay as the deflocculent. The finished coating mixture contained 62% solids by weight, the starch-to-clay ratio being 15 to 100 on a weight basis. Five per cent of soap, based on the starch, was used.

The first part of the table shows the variations in viscosity and flow which can be obtained by blending a low viscosity special coating dextrin with a low viscosity oxidized starch. It will be noted that as the amount of oxidized starch is increased the degree of thixotropy is increased, resulting in an improvement in leveling index. However, increasing the amount of oxidized starch increased the low shear viscosity as measured on the Brookfield and increased the plastic viscosity. Because of this high viscosity at low shear, it

Table III. Effect of Soap on Flow Properties of Coating Mixtures

Adhesive—Special Coating Dextrin

Type of soap	Soap, %	Brookfield viscosity, cp. Fresh	Brookfield viscosity, cp. Aged 24 hrs.	Plastic viscosity 500 r.p.m.	Plastic viscosity 1000 r.p.m.	Leveling index
Hard, sodium base	3	1000 (20 r.p.m.)	1400	1.44	1.29	0.17
Hard, sodium base	5	1340 (20 r.p.m.)	1300	1.80	1.49	0.30
Ammonium stearate	1	500 (20 r.p.m.)	800	1.04	1.02	..
Ammonium stearate	2	520 (20 r.p.m.)	720	1.16	1.18	..

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Table IV. Effect of Soap on Flow Properties of Coating Mixtures

Adhesive—70% Special Coating, Dextrin 30% Oxidized Starch

Type of soap	Soap, %	Brookfield viscosity, cp.		Plastic viscosity		Leveling index
		Fresh	Aged 24 hr.	500 r.p.m.	1000 r.p.m.	
Hard, sodium base	3	2400 (20 r.p.m.)	4100	2.05	1.72	0.28
Hard, sodium base	5	2860 (20 r.p.m.)	4400	2.41	1.94	0.35
Ammonium stearate	1	2340 (20 r.p.m.)	4900	2.30	1.87	0.33
Ammonium stearate	2	2740 (20 r.p.m.)	4260	2.55	2.05	0.35

is doubtful that the coating made with 100% oxidized starch would be commercially usable at the solids used in these experiments. Oxidized starches are, of course, widely used commercially for coating but generally in a lower range of solids.

Since there is considerable interest in set-back characteristics of coating mixtures, an attempt was made to measure this characteristic by the use of low shear viscosity figures for the coating mixtures when fresh and after standing for 24 hr. It can be seen in Table I that setback of the coating mixture becomes progressively greater as the amount of oxidized starch in the coating mixture is increased. The special coating dextrin used in this study was designed for a minimum of setback and that is the reason for the good results obtained in regard to this characteristic. Setback is important from the standpoint of ease of handling the coating mixture. In some cases not all of the coating is transferred to the paper and part of it must be returned by pumping to the storage tank, and in such a case ease of handling is necessary.

Also depicted in Table I are coatings made with a white dextrin of medium viscosity and one in which cornstarch was enzyme-converted. This dextrin produced almost identical plastic viscosity and leveling index values as that obtained from the above special coating dextrin, but the original viscosity is higher and there is more setback. This is the result of the different methods by which the two dextrins were manufactured.

Table II illustrates the difference in flow properties which can be obtained with the same types of starches used above but prepared with only 3% of soap. Here the improvement in leveling index as the ratio of oxidized starch is increased is even more pronounced. Although there is little difference in the low shear viscosity of the first three coatings in this table compared to the same coatings in Table I, there is a decided contrast in the leveling index in each case.

In the case of the medium viscosity white dextrin, while the plastic viscosity has been increased very little, there is a decided improvement in leveling index. The low shear viscosity has been increased only slightly but the setback of the aged coating has increased substantially. The enzyme-converted starch coating in this table has a considerably lower Brookfield value than its counterpart in Table I and exhibits improved thixotropic qualities as evidenced by the lower plastic viscosities and high leveling index.

Effect of Soap

Table III is concerned with coatings sized with 15% of the special coating dextrin. It can be seen that increasing the soap from 3 to 5% has brought about sufficient improvement in the leveling index to raise the coating from the inoperable

Table V. Effect of Soap on Flow Properties of Coating Mixtures

Adhesive—50% Special Coating Dextrin, 50% Oxidized Starch

Type of soap	Soap, %	Brookfield viscosity, cp.		Plastic viscosity		Leveling index
		Fresh	Aged 24 hr.	500 r.p.m.	1000 r.p.m.	
Hard, sodium base	3	8200 (20 r.p.m.)	60,000	3.02	2.26	0.47
Hard, sodium base	5	7700 (20 r.p.m.)	13,000	3.21	2.48	0.42
Ammonium stearate	1	4160 (20 r.p.m.)	8,240	2.91	2.20	0.47
Ammonium stearate	2	5300 (20 r.p.m.)	10,240	2.55	2.03	0.37

class to one which presumably would produce a pattern-free application. This improvement is due to the increase in thixotropy effected by the additional 2% of soap. Again the special coating dextrin has such good resistance to setback that the viscosity of the coating mixture is almost unchanged after standing for 24 hr.

Table IV shows that a smaller amount of soap is required to obtain a satisfactory leveling index when oxidized starch is used in a blend with the special dextrin than when the dextrin is used alone. The greater amount of soap again provided the increase in this coating made with a blend of 70% of the special

Table VI. Effect of Soap on Flow Properties of Coating Mixtures

Adhesive—Enzyme-Converted Starch

Type of soap	Soap, %	Brookfield viscosity, cp.	Plastic viscosity 500 r.p.m.	Plastic viscosity 1000 r.p.m.	Leveling index
None	0	4,540 (20 r.p.m.)	2.90	2.35	0.34
Hard, sodium base	1	5,500 (20 r.p.m.)	2.19	1.81	0.30
Hard, sodium base	3	7,600 (20 r.p.m.)	2.89	2.22	0.44
Hard, sodium base	5	11,200 (20 r.p.m.)	3.05	2.39	0.40
Ammonium stearate	1	5,400 (20 r.p.m.)	2.69	2.21	0.32
Ammonium stearate	2	7,000 (20 r.p.m.)	2.58	2.12	0.32

Table VII. Effect of Stabilizers on Starch Solution Viscosity

Adhesive—Enzyme-converted starch

Liquefying agent	Stabilizer, %	Brookfield viscosity, cp. Fresh	Brookfield viscosity, cp. Aged 2½ hr.	Viscosity increase, %
None	0	15,500 (10 r.p.m.)	19,000	22.6
Dicyandiamide	5	11,200 (10 r.p.m.)	13,000	16.1
Dicyandiamide	10	9,200 (10 r.p.m.)	10,000	8.7
Dicyandiamide	15	8,400 (10 r.p.m.)	8,800	4.8
Dicyandiamide	20	7,100 (10 r.p.m.)	7,500	5.6
None	0	16,700 (10 r.p.m.)	18,400	10.1
Ammonium thiocyanate	5	14,000 (10 r.p.m.)	14,500	3.1
Ammonium thiocyanate	15	7,800 (10 r.p.m.)	9,400	20.5
Ammonium thiocyanate	20	6,100 (10 r.p.m.)	7,500	22.9

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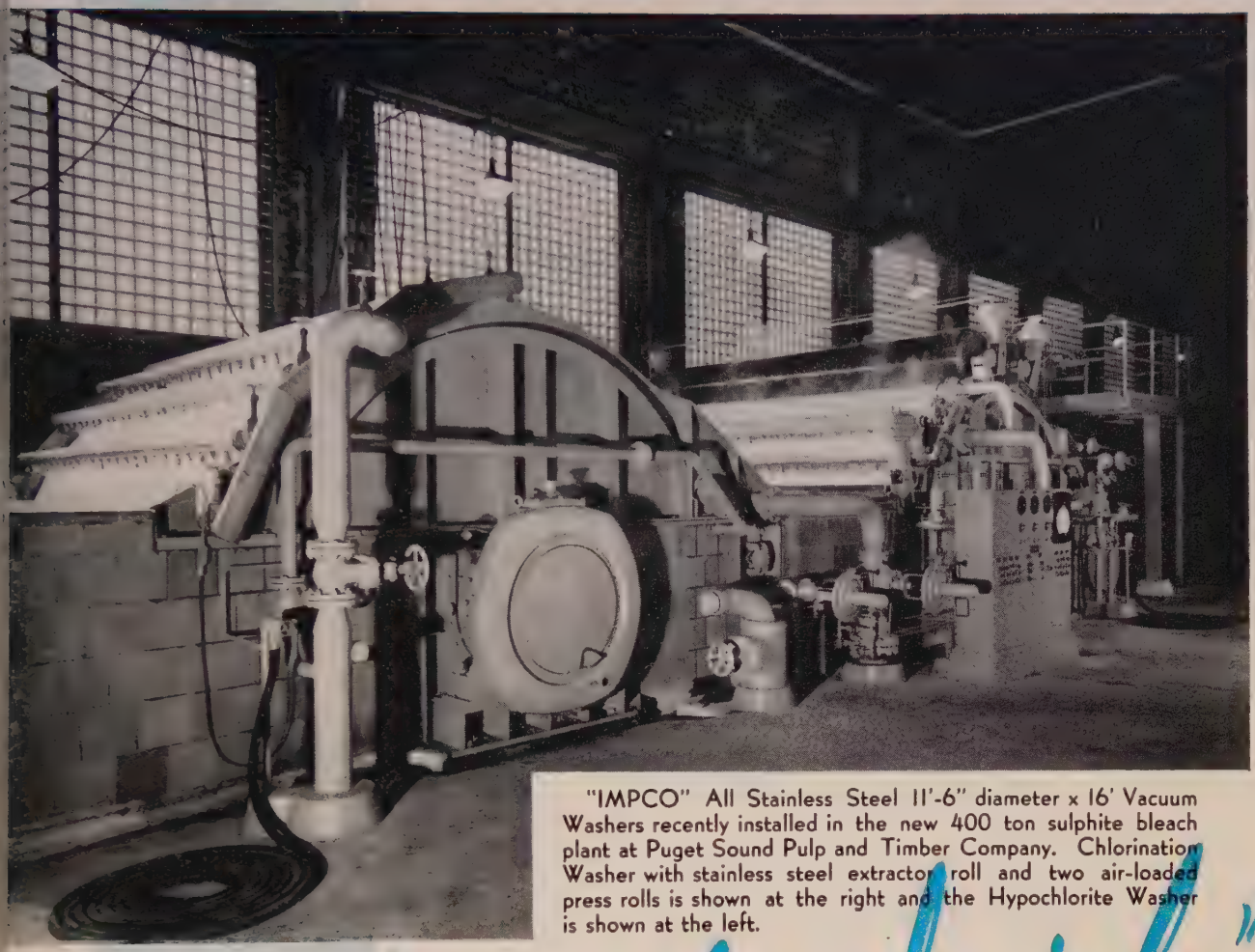

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Table VIII. Effect of Stabilizers on Coating Viscosity

Adhesive—Enzyme-converted starch

Liquefying agent	Stabilizer, %	Brookfield viscosity, cp. Fresh	Aged 2 hr.	Aged 6 hr.
None	0	5100 (10 r.p.m.)	6700	10,000
Dicyandiamide	5	5000 (10 r.p.m.)	5700	7,500
Dicyandiamide	15	4200 (10 r.p.m.)	4800	7,000
Dicyandiamide	20	3100 (10 r.p.m.)	3700	5,500

coating dextrin and 30% oxidized starch as the adhesive. This coating is influenced much more by ammonium stearate than the coating made with dextrin alone. The good aging properties of the dextrin were again noted.

Table V illustrates the properties of a coating made with a mixture of 50% special coating dextrin and 50% oxidized starch as the adhesive. This combination again produced a coating mixture of high original viscosity and increases set-back qualities. In all cases the leveling indices are excellent and provided that other considerations did not prohibit, each of these coating mixtures could be considered operable. It is apparent that this particular blend of binders has a critical soap concentration beyond which the leveling properties become poorer.

It is not too general a statement to say that it is virtually possible to build almost any desired characteristics into an enzyme-converted starch by controlling the conversion procedure and applying additives judiciously. Table VI is, to a limited extent, an illustration of this statement. Here a converted starch with reasonably good thixotropic and leveling qualities has had these qualities enhanced by the addition of soap. The addition of 3% of hard soap has benefited the leveling index substantially without altering the low shear viscosity too severely and with little effect on the plastic viscosity. This particular coating formula was improved to a much lesser extent by the addition of ammonium stearate.

Effect of Stabilizing Agents

To illustrate the stabilizing effect of certain chemicals on enzyme-converted starch solutions, a substantial quantity of starch was converted at 30% solids, cooled to 170°F., the viscosity recorded, and additions of various percentages of two different stabilizers (based on starch weight) made immediately. The viscosities were recorded as soon as possible. After allowing the solutions to stand for $2^1/_2$ hr. with the temperature maintained at 170°F., the viscosities were again measured. These values are illustrated in Table VII.

Since there is a graduated reduction in original viscosity caused by the increase in amount of chemical added, a factor which has been designated as "per cent viscosity increase" and is the ratio of the increase in viscosity to the original viscosity was devised to evaluate the efficacy of the stabilizers. For this particular type of enzyme conversion it appears that the addition of 5% of ammonium thiocyanate produces the minimum of viscosity increase while causing only minor reduction in the original viscosity. In addition to the ammonium thiocyanate mentioned, dicyandiamide was also tried. There are other effective chemicals, such as sodium thiocyanate, sodium acetate, and urea which might have been investigated. The effectiveness of these stabilizers will vary somewhat with the circumstances of conversion of the starch.

In order to determine the effect of stabilizing agents on the prepared coating, starch solutions made in a fashion similar to that described above were used as the adhesive for 60% solids coatings. The results of one particular set of experiments are recorded in Table VIII. Here again the effect of 5% of stabilizer produced the best reduction in viscosity setback consistent with the desired fresh viscosity in a reasonable time period of 2 to 6 hr.

Table IX. Stability of Starch Solutions

Starch	Brookfield viscosity, cp. Fresh	Aged 2 hr.	Aged 4 hr.
Enzyme-converting starch X	12,000 (10 r.p.m.)	23,500	26,000
Enzyme-converting starch Y	12,000 (10 r.p.m.)	11,500	11,300

Starches for enzyme conversion appear to produce solutions which differ in viscosity set-back characteristics. The results of one experiment is depicted in Table IX. Under the particular set of circumstances involved in this experiment it will be seen that the enzyme-converting starch designated by Y maintained its original viscosity during a 4-hr. period while the enzyme-converting starch designated by X showed a viscosity increase amounting to 100%.

CONCLUSIONS

In summing up it should be re-emphasized that this study has been greatly simplified. The chief points of interest in starch modification and their effect on rheological properties of coating mixtures have been outlined. It has been shown that the difference between an inoperable coating color and an acceptable one is often a matter of a simple change in the properties of the starch being used or a minor change in coating color formulation.

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Water-Resistant Starch Coatings

W. C. BLACK

In recent years there has been a constantly increasing interest shown in methods of giving to starch coatings the water resistance desirable or necessary to satisfy certain end-uses. Starch adhesives have long been useful in paper coatings, but the finished sheet has not possessed appreciable water resistance so that the housewife's fingers picked the coating off a soap powder container when she picked it up with wet hands, or the lithograph or offset press operator was faced with frequent clean-ups because the coating on the paper being printed picked off on the moistened blankets or rolls applying the ink to the sheet. It was in many instances necessary to use other types of adhesives which developed the necessary water resistance, but which were more expensive and from time-to-time in limited supply.

Chemically, starch may be classed as a polysaccharide or polyalcohol and the molecules are in the form of long chains of glucose or simple sugar units. The many —OH groups on this long chain molecule are hydrophilic in nature so that when heated in water their association tendency for the water is great enough that internal molecular attractions in the granule are overcome and the starch becomes "cooked" or "dispersed." A resulting increase in clarity, viscosity, etc., is usually observed. When this water is removed, as by drying, associative forces between the molecules are again re-established and a film results. This film, however, is quite sensitive to moisture so that on being remoistened it has a strong tendency to again disperse in water and rapidly becomes soft and useless as an adhesive.

Many efforts have been made to decrease the water sensitivity of starch films and coatings and have followed a variety of approaches. A mention of a few of these follows.

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ADDITION OF WATER-RESISTANT MATERIALS

On the theory that the properties of a film of a mixture of two compatible, film-forming materials are somewhere between the properties of films of each ingredient alone, numerous additives have been incorporated in starches to improve their properties. Rubber latices, both natural and synthetic, have been found effective as regards wet-rub improvement and the latter has attained appreciable acceptance for some uses (*1*). In effect, the starch acts somewhat as a diluent or extender for the latex which must be present in amounts of at least 25 to 50% on the starch to give appreciable wet-rub resistance. Such materials have the advantage of producing a flexible sheet which is desirable in some instances, but the disadvantage of being relatively high in price so that coatings containing them cannot be classed as inexpensive.

Polyvinyl alcohol has also been suggested for use in making starch films more water resistant (*2*), especially in conjunction with aldehydes or certain resins to aid insolubilization. Such uses have merit in certain types of sizes and adhesives. In the usual paper coatings, however, the viscosity effects of the PVA are such that the starch content or the total solids of the color must be reduced enough to maintain a usable viscosity that the advantages are largely minimized or eliminated. Economically, too, we believe the expense of the added PVA can be more efficiently utilized by substituting a greater amount of starch and resin as noted below.

A number of materials have been suggested as possible additives to starch films and coatings for the purpose of keeping the film from wetting so that its disintegration on contact with water will be retarded. Wax emulsions, rosin emulsion, etc., are effective to a degree but frequently cause side-effects which are undesirable. Other materials which attach a fatty molecule to the film, as stearato chromic chloride, have also been evaluated (*3*). We have not found them effective when mixed with the coating color although a surface application gave a degree of transitory repellency. This particular material also tends to impart a color to some coatings which probably would be objectionable in many instances.

ADDITION OF MATERIALS WHICH COMBINE WITH THE STARCH TO GIVE A PRODUCT WITH CHANGED REDISPERSING PROPERTIES

These materials may or may not have filming properties in themselves, but have the ability to combine chemically with the starch molecule and by so doing change its redispersing properties. Two mechanisms by which these changes are brought about have been proposed: (1) the material forms cross-links or bridges between adjacent starch molecules with the new reinforced molecular structure being rigid enough to resist the swelling which normally takes place on rewetting. Access to additional water is prevented or at least retarded and the rate of disintegration is reduced. (2) The material added merely attaches itself to certain hydrophilic groups on the starch molecule and blocks them off or makes them unavailable for association with water. Such blocking of a relatively few key groups could conceivably prevent hydration. Which of the two proposed mechanisms is correct is immaterial for the purposes of this discussion, but either will explain why a small proportion of certain additives so materially changes the redispersing properties of starch films. Materials of this nature which have attained appreciable commercial usage are of two types.

Metallic Salts

Certain multivalent-metal salts having groups which tend to associate with the —OH groups on the starch molecule are examples of such materials. Under properly selected conditions some chromium and antimony compounds unite with starch to form more or less irreversible combinations which have increased resistance to redispersion in water (*3*, *4*). Potassium pyroantimonate is currently being used for this purpose in paper coatings, and 5 to 10% of this salt (on the starch basis) appreciably improves the water-resistance of starch-containing coatings provided the correct combination of drying (time-temperature-moisture) and aging conditions are available (*5*). As the material is relatively expensive, economics might in some cases limit its applicability. It has, however, the advantage that the optimum pH for water-resistance development is slightly above neutral and in a range desirable for some types of coatings and pigments.

Aldehydes and Aldehyde Resins

Another type of material which combines with starch and so influences its redispersing tendencies is the aldehydes and aldehyde resins. For many years the insolubilizing effect of formaldehyde on starch has been known and recently much work has been done on the similar effect of glyoxal (*6*) as well as a variety of aldehyde resins including melamine-formaldehydes (*7*, *8*), urea-formaldehydes (*9*), ketone-aldehydes, resorcinol-aldehydes, etc. Some of these resins are now being used in large tonnages to insolubilize starch adhesives used in the fiber container and paper bag industries. They enable the manufacture of items not possible with ordinary starch adhesives and at a cost which is attractive compared to acceptable adhesives of other types. To our knowledge successful starch coatings have been prepared only with the urea-formaldehyde or melamine-formaldehyde type of resins and our work has been devoted principally to the former largely because of its operational and economic advantages. The behavior of the two resins with starch is quite similar except that the melamine-formaldehyde is somewhat more effective on an equal weight basis and its films are more stable under certain conditions.

FACTORS AFFECTING STARCH-RESIN COATINGS

As can be imagined the use of such starch-resin adhesives in paper coatings imposes an entirely new set of conditions and limitations on the adhesive mixture and the balance of this discussion will be concerned with some of the factors which have been found to have a bearing on the operational performance of the coating color and on the development of water resistance in the applied films.

The combination of the urea-formaldehyde resins with starches responds in general to condition changes much the same as does the polymerization reaction of the resin alone. Increasing the temperature, time of contact, or concentration (of either starch or resin), or lowering the pH tends to advance the degree of insolubilization. The idea, of course, is to balance the different variables against the machine conditions so that a coating having acceptable water resistance results at the time the sheet is to be used or tested.

It appears that a coating containing certain proportions of starch, resin, and pigment has a certain potential wet-rub resistance and the degree to which the actual wet-rub resistance approaches this potential or maximum depends on the following factors.

Cooking Procedures

It is possible to bring about a combination of the starch and resin both during the cooking procedure before the mixture is added to the clay slip (*10*) and also in the coating film after application, as during the drying operation or during aging of the finished sheets. Such a possible preliminary combination of starch with the insolubilizing agent, as during cooking, has also been noted in the use of antimonates (*5*). This preliminary or initial starch-resin combination can be still further promoted by the acidification of the cooked material while still near the peak temperature (*11*, *12*). Depending on the concentrations, type of starch used, etc., a viscosity rise may result on acidification, but on continued heating this soon decreases to a usable level. When the drying or aging

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conditions are not sufficient to develop all of the desired insolubilization, it is, therefore, possible to supplement the combination on drying with a degree of combination brought about during preparation. Such prior combination with resin appears effective when resin solids up to about 10 to 15% on the starch basis are employed. Cooking the starch with more than this amount of resin appears to have no advantages so that, if say 20 to 30% resin is found necessary to obtain the desired wet-rub resistance, the balance of the resin should be added to the finished color.

Cooking the starch in the presence of resin changes the so-called water-holding properties of the starch, as will be mentioned later, and usually gives a color having a higher viscosity than one with the same composition but in which all of the resin solids are added to the finished starch-pigment color. The "reacted" starch-resin mixtures (acidified while hot) cause a still higher final color viscosity. Although frequently desirable for promotion of maximum insolubilization, these procedures in which a prior partial combination of resin and starch is effected are often limited in application because of this viscosity effect even though a thinner or less viscous starch may be substituted.

Effect of Time

The time the sheet is in the drier as well as the temperature the sheet reaches in the drying section is usually fixed by other considerations so that for a given operation little variation in these factors is possible. Normal changes in machine speed as the sheet weights are changed may be sufficient to increase or decrease this curing time enough to cause noticeable variations in the degree of insolubilization obtained with the same coating color. As an extreme case the hour or two drying cycle in the case of wet-end coated wallboards enables the obtaining of a much higher degree of wet-rub resistance with a given color than is possible on more rapidly dried paper or carton stock.

In some instances a considerable portion of the final wet-rub resistance is developed as the coated sheet is aged prior to use. The paper rolls frequently remain warm for a period of time which contributes materially in some instances to the starch insolubilization. Aging for a few days to a week is often necessary to produce a sheet having the desired properties.

Concentration Effect

The concentration or solids content of a coating color is a factor in determining the resin necessary for obtaining a given wet rub. Possibly because of the changed penetration characteristics of the higher solids more viscous colors, the adhesive requirement for a given pick test or abrasion test is usually lower for these colors. A 60% solids color may require but 15% starch on pigment to produce a given pick test while a similar color at 40% solids may require upward of 20% starch to obtain the same pick. The resin requirement for a given wet-rub development apparently follows a similar pattern so it has been found convenient and reasonably accurate to express the resin concentration necessary as a percentage of the starch present.

pH Adjustment

Probably the most important factor controlling the insolubilization of starch-resin films is the pH of the film when it is subjected to the previously mentioned factors of time and temperature during drying and aging. As with the resins alone, lowering the pH of starch-resin combinations accelerates the insolubilization reaction. We have found the preferred range for coatings to be from 4.0 to 6.0 pH. Above 6.0 the rate is usually so slow as to be unsuitable, while below 4.0 there is the danger of poor aging characteristics especially at conditions of elevated humidity and temperature. A difference of but a few tenths in the pH of a coating may materially affect the resulting insolubilization in some instances, whi with other coatings dried in different ways or possibly i slightly different pH ranges a similar pH change will have n apparent effect.

The desired acidic conditions in the coating film can b obtained by the use of either direct catalysts, such as aci which lower the pH immediately, or by the use of so-calle latent or delayed catalysts. Th latter are usually ammoniu salts which on drying combine with the free formaldehyd always present in, or released by the resin to liberate a fre acid which lowers the pH. Almost any acid or acidic salt suitable for pH adjustment of ordinary starch-resin adhesive When used with clay suspensions, however, many of the acids or salts have a flocculating action on the clay with th resulting viscosity increases making the color unusabl Syrupy metaphosphoric acid has been found quite satisfactor for lowering the color pH and has a minimum flocculatin effect on the clay. It is preferred that it be added to th finished color as a freshly made 10 to 20% dilution in wate

It is also necessary, obviously, to select a clay which has s minimum sensitivity to lowered pH's and that the dispersan used be one that retains its dispersing or viscosity-lowerin properties at pH's in the desired 4.0 to 6.0 range. The amou of dispersant used may have to be increased somewhat t obtain the optimum viscosity at the desired lowered pH Even at the desired low pH's we have seldom experience difficulty from viscosity increases on aging of the colors.

In certain instances where it is undesirable to lower th color pH to the desired level at the time of application, th lowered pH necessary for curing during drying may be obtaine by the use of the latent catalysts mentioned. From 10 t 50% on the basis of the resin solids present is usually necessar depending on conditions and choice of resin. Among th most satisfactory are the phosphates which have a minimun flocculating effect on the clay, as diammonium phosphat ammonium hexametaphosphate, etc.

In other instances it has been found desirable to lower th applied color pH by the separate application of the catalys either under or on top of the applied color. For example, a alum wash applied to the dried surface of a coated wallpape effectively insolubilized the starch-resin-clay coating use Similarly, a calender application of alum solution prior t coating was necessary in one instance to enable the develo ment of the desired water resistance on a coated board. I each case starch was added to the acid wash to prevent undu penetration into the sheet. The pH of the sheet being coate is also a factor in the wet-rub development. For exampl drawdowns made with a starch-resin-clay coating color havin a pH of below 4.0 would not develop noticeable wet-rub resist ance when applied to the bottom side of an originally aci sheet to the top side of which a mildly alkaline protein coat ing had already been applied. When applied to the sam sheet without the alkaline top-side coating, excellent wet rul was developed.

"Water-Holding" Ability of Starch

In water-resistant starch coatings, as well as in other type of starch coatings and in starch sizes, there is another starc characteristic which is of an importance which we believe i either not generally recognized or is underestimated. Thi may be described as its "water-holding" power and deter mines to a large degree the penetration of the adhesive o size into the sheet. If similar pastes are made by cooking tw starches of about the same fluidity, say a thin-boiling starc and an oxidized starch, and a few drops of each paste are pu on the surface of a piece of filter or other adsorbent paper, th migration rate of the starches into the paper may be quit different. In a short time there will be observed a wette ring of considerable size around the drop of thin-boiling starc paste, while the oxidized starch will exhibit a negligible rin or in some instances may not even wet through the filte

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paper. This same water-holding ability is carried through to a degree in coating colors made from these same starches and largely governs the adhesive penetration into the sheet as well as the adhesive distribution in the dried film of coating. It is apparently generally recognized that this adhesive migration in the sheet and its distribution in the coating itself are of importance in determining the pick or strength of a coating (*13*). Similarly, it is of importance in governing the wet-rub resistance of a coating especially as the resins used are of a smaller molecular size than the average starch fragment and in part tend to migrate along with the so-called free or unbound water. The use of a starch, therefore, which tends to keep the liquid portion of the coating, along with its dissolved starch and resin, within the physical limits of the coating layer should produce a final coating having a higher strength both in the wet and dry states. This is what appears to happen. As mentioned previously, the oxidized starches are superior in this respect along with some dextrins and some of the low substituted starch derivatives which are now available.

In work directed toward the investigation of the starch distribution in the dried coating layer and in the adjacent fiber layers there frequently is no mention made of the type of starch used. For the above reasons any distribution data obtained experimentally would present the picture for but a single starch and might be widely in error for starches with different hydration properties.

It is apparent that sheet characteristics which also affect the water migration from the adhesive would be a factor in determining the characteristics of the finished coating. This has been demonstrated many times in practice as regards the dry strength of the coatings and it has been found true also as regards wet-rub development. A soft-sized or highly porous sheet will usually show a lower wax pick and inferior wet-rub resistance. On the other hand, a sheet that is extremely hard sized will not wet sufficiently for the coating to form a bond with the fibers on the surface and poor adhesion results, although the water resistance and strength of the coating layer itself may be good. Especially with those starches having relatively poor water-holding properties, the procedure of cooking the starch in the presence of the resin tends to improve this characteristic for both the paste and the resulting color. This feature may be a contributing factor in the improved wet-rub resistance frequently obtained when such a procedure is followed.

Selection of Resin

A wide variety of urea-formaldehyde resins are available from various manufacturers and many of them have proved satisfactory in use. Improved wet-rub resistance can be obtained by using the simplest material, dimethylol-urea. However, it is usually more convenient and also somewhat more effective to use a more highly polymerized product which may still be diluted in all proportions with water and which is usually sold in the form of a liquid containing 50 to 65% solids. While the urea-formaldehyde ratio and the degree of polymerization may vary somewhat between the various products offered, the results obtained are surprisingly uniform. Some resins available apparently contain salts which have a flocculating or thickening action on clays. While such resins are widely used in starch-resin adhesives, they are unsuitable for coatings and it is always desirable to laboratory check an unknown or new resin for this feature.

Selection of Starch

Frequently several types of starches are available which will give acceptable results in starch-resin coatings. Under certain conditions, however, some of them may have pronounced advantages over others so that each application must be considered individually and the most desirable starch selected, frequently after considerable laboratory or actual mill evaluation. The relative importance of the starch's water-holding ability should be considered in association with the sheet to be coated and the equipment to be used. The suitability of the color's flow properties, such as degree of thixotropy, etc., as affected by different starches, for the type of equipment to be used must also be considered. The tendency or ability of different starches to combine with resins, as for insolubilization, varies considerably even at a given fluidity or degree of modification. Depending on the leeway available in the coated sheet the selection of a more easily insolubilized starch might permit the use of less resin or the development of otherwise unobtainable wet-rub resistance.

Probably the most important factor in starch selection is that it must give a color viscosity in the desired range when used in a given proportion in a color of a given solids content. For a particular type of starch the viscosity of a given color varies as the viscosity of the cooked or pasted starch incorporated in it. Because of the different clay-dispersing abilities of different types of starch, however, colors made using starches of the same fluidity but of different types or methods of preparation might be widely different.

As both starch pastes and starch-clay coating colors are thixotropic to varying degrees, the results obtained on different types of viscometers may not be at all comparable. Starch A may have an indicated viscosity somewhat higher than starch B of a different type on one viscometer, but because of different flow properties its viscosity on another viscometer which imposes different conditions on the paste during testing may be indicated as lower. As the use conditions are again so different from those to which the color is subjected in the usual viscometers, the performance of a color cannot, therefore, be predicted solely on the basis of an indicated viscosity. Viscosity data obtained in any way are useful only in comparing starch pastes of a given type or the colors made from them. In comparing starch colors with, for example, protein-containing colors, viscosity data may lead to very erroneous machine performance predictions. We have several times successfully run starch-resin colors on both air-knife and roll-type coaters that were way beyond the maximum indicated viscosity found suitable for the ordinarily used protein coating color. It is of prime importance, therefore, to evaluate a starch or starch coating color in terms of the viscosity relationships which have been established for other starches or colors having similar flow characteristics.

Other Procedure Variations

A number of operational variations have been attempted with water-resistant coatings in an effort to obtain improved results or reduced costs. The addition of starch to the base sheet, as by furnish addition or tub or calender sizing prior to coating, has been found in some instances to appreciably lower the adhesive requirements of the coating itself. Also it is not uncommon in mills having facilities for applying more than one coat per side to put a protein or starch-resin water-resistant coating over a non-water-resistant somewhat cheaper starch-clay base coat. Depending on the use requirements such a dual application is often quite satisfactory. Several mills are currently experimenting with a procedure in which a starch-clay coating, that may be utilized as this undercoat, is applied to the sheet by the usual size press. Preliminary results have been encouraging. Another approach for some uses is a very light surface application of resin and starch as a wash over a non-resistant starch-pigment coating. The wet-rub development is excellent and the added cost is negligible, but unfortunately the surface so treated is so dimensionally stable that changes in moisture content cause excessive curling which limits the use of the product.

Evaluation of Results

Two of the undecided questions at many mills are: "how much wet-rub resistance is necessary?", and "how shall it

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be measured?" Most of the interest shown in starch-resin coatings has been by mills who feel a need for a product having improved wet-rub resistance as compared with straight starch coatings, but not necessarily as much as protein coatings, and at a cost materially less than the latter. Although it is admitted by many coaters that the ultimate in wet-rub resistance is not required by many customers, coated samples are almost universally tested by crude but rigorous methods. A few drops of water are poured on the sheet and after a few seconds are rubbed into the surface with the maximum pressure the thumb or finger will stand. Any pigment removal usually classes the sheet as unsuitable. In another test the thumb is pressed with considerable pressure onto the coated surface which had been just moistened with a few drops of water. A rapid upward pulling-away of the thumb should not cause any pigment to adhere to it. These are rather drastic tests when it is considered how lacking in water resistance the original starch coatings are, yet it is expected that even the "slightly improved" coatings desired should pass them. Results of both the above tests frequently vary widely depending on how much water is used, how many seconds have elapsed between the initial contact of water with the sheet and the completion of the test, and on how much pressure is applied to the rubbing or pressing finger. A variation of the above rubbing test has been proposed in which the coating removed on each finger stroke is partially transferred to the surface of an adjacent sheet of black paper (*1*, *9*). A comparison of the resulting white smears gives numerical results, but the test is still subject to human variation in operation and interpretation. A test has been proposed in which the coating actually removed by a controlled rubbing of the immersed sample is estimated on a turbidimeter (*14*). This test is of value in comparing sheets having an equal added coat weight for an end-use where the coated sheet is completely wetted. Various scrubbing tests have also been devised and are in use in the wallboard and wallpaper industries, but are usually much too severe for the average coated paper or board. Regardless of the evaluation procedure used it would appear desirable to interpret the results more in the light of the needs of the ultimate consumer than is presently done. Very frequently this same consumer is willing or anxious to slightly change his procedures to lessen the demands on the product—especially if he will save money by so doing.

CONCLUSION

It is believed that properly prepared starch-resin-pigment coatings will produce a coated sheet satisfactory to the needs of a considerable proportion of the consumers requiring a degree of wet-rub resistance. As so many factors enter into the development of this wet-rub resistance, it becomes necessary to consider each application separately and to select the materials, their proportions, and the procedures used so that the desired sheet characteristics may be obtained within the limits of the available equipment and its operational requirements.

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Dextrines as Coating Adhesives

T. A. WHITE, L. J. WOOD, W. F. SMART

STARCH-base adhesives have been used for coating purposes for many years. This symposium, of which this paper is a part, is designed to review and discuss the various types of starch products which have been found of use as coating adhesives. It is the object of this paper to review particularly those materials known as dextrines. In order to present the case for dextrines it is necessary to compare dextrines with the other types of starch products. Oxidized starches and enzyme-converted starches have their own characteristics of value for coating purposes. No attempt is made here to review their advantages but they are mentioned in order to bring out more clearly the characteristics of dextrines. It is felt that a more complete understanding of the subject may be obtained in that way.

TYPES OF DEXTRINES

Before reviewing the use of dextrines for coating purposes it might be helpful to review briefly the various types of dextrines as materials There are, in general, three types of dextrines commonly referred to as British gums, white dextrines, and yellow dextrines. Sometimes oxidized starches are referred to as "gums", but they should not be confused with British gums. All of them are made by roasting starch in a piece of equipment known as a cooker. There are three variables which must be controlled carefully in order to manufacture dextrine products. They are the temperature at which the roasting is done, the time of roasting, and the pH of the starch which is roasted. By varying the conditions of roasting it is possible to manufacture converted starches with a wide variety of physical characteristics.

British Gums

Of all the materials manufactured under the general classification of dextrines, the British gums offer the widest range in viscosity. They vary in color from an off-white to a rather dark brown. Some of the less converted types are almost as thick cooking as the raw starches. The more converted types are almost as thin cooking as the yellow dextrine. They are all manufactured by roasting starch at higher temperatures. In some cases they are roasted with no acid present and in some cases it may be done on the alkaline side. Other British gums are roasted with some acid present, but in general the pH at which British gums are roasted will be higher than would be the case with yellow dextrines. The main factors in the conversion of British gums are temperature and time and these produce products which form cooked pastes of better than average stability to viscosity changes with age. They are not as pasty. In the adhesive field they are referred to as filmy, which means that in texture they have a certain amount of cohesiveness so that on spreading out the cooked paste a more uniform film may be formed. The filminess, sometimes referred to as higher water retention, prevents its penetration into paper, for example, as readily as would a pasty type of product. It tends to coat over the surface more readily. British gums would probably be more useful as coating adhesives if the colors were not so dark. The lighter colored British gums generally have higher viscosities than can be used successfully in pigment coatings In cases where the color would not be objectionable, British gums offer interesting characteristics for pigment coatings.

White Dextrines

Like British gums, white dextrines are roasted in cookers;

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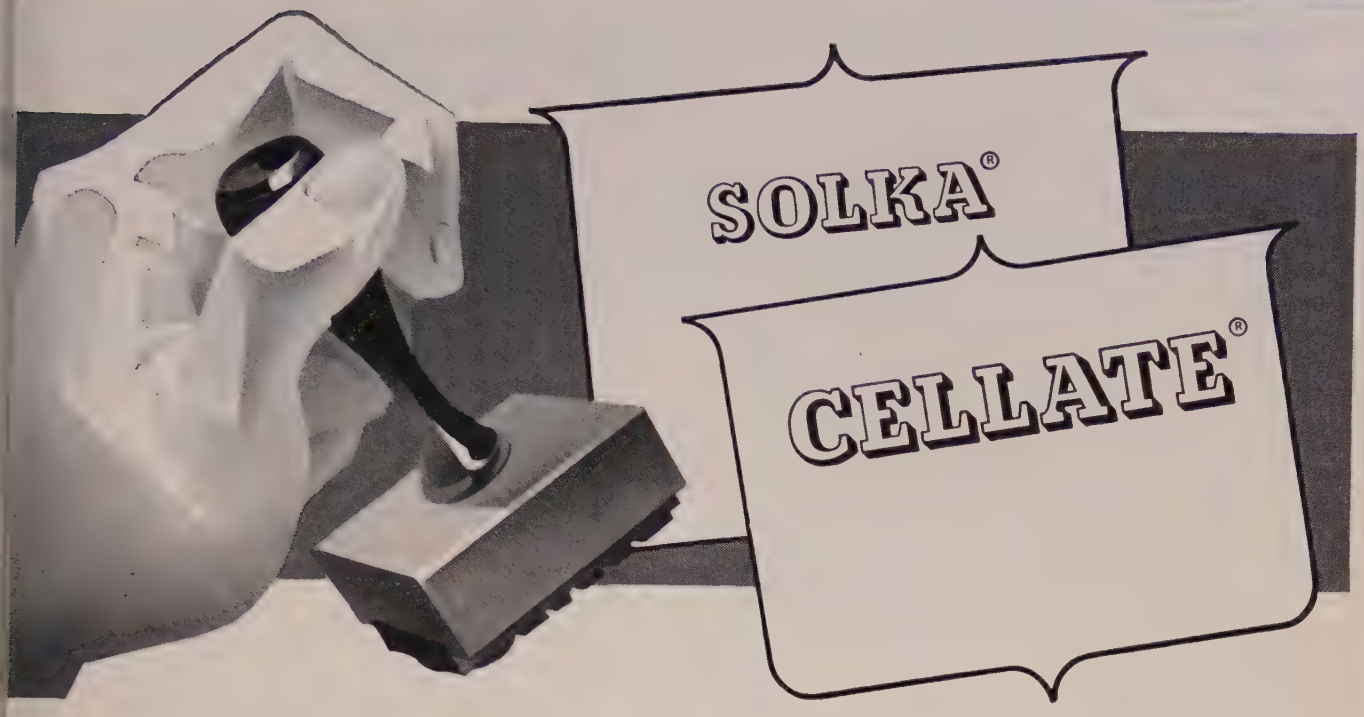

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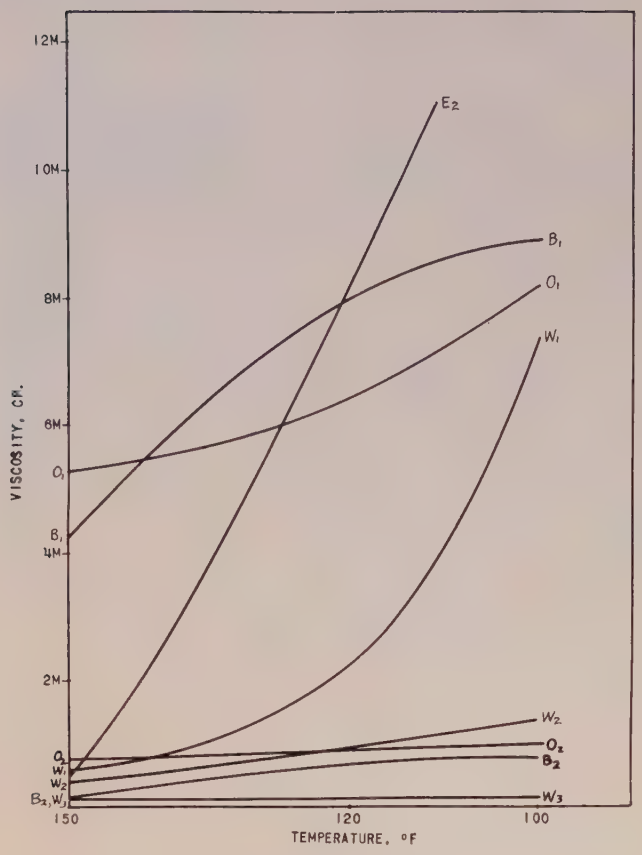

Fig. 1. Temperature stability of various starches at 2 r.p.m. and 25% solids

but the roasting is done at a much lower temperature and in the presence of acid. The pH at which white dextrines are roasted is generally the most acid for all of the dextrine types. Unlike British gums, white dextrines are light colored and, in general, are white to only slightly off-white in color. Their cooked and cooled solutions tend to set to soft pastes in a relatively short time. They do not have the filmy characteristics of British gums. White dextrines are manufactured in a variety of viscosity types. The lower viscosity, or more converted, types are generally slightly off-white in color and have a viscosity equivalent to that of yellow dextrines. The thinnest British gum will not normally be quite as low in viscosity as the thinnest white dextrine. The strength of the more highly converted white dextrines, as is the case with all highly converted products, is low. The lesser converted types of white dextrines give good strength and are manufactured in a variety of viscosities which are of interest for both off-machine and machine coating. In general, white dextrines can be converted further with lower viscosities and suitable adhesive strengths, than can be done by other means of conversions. Solutions of white dextrines tend to thicken or paste up rather quickly as compared to British gums or yellow dextrines, and hence are referred to as pasty types. They tend to penetrate porous paper more readily than do filmy, stable types, with higher water retention.

Yellow Dextrines

Less acid and more heat are used in roasting yellow dextrines as compared with white dextrines. On the other hand, more acid and less heat are used than in the manufacture of British gum. That is the general procedure but, of course, a variety of combinations can be employed in order to manufacture a type of product with the characteristics desired for any given purpose. This holds true also for white dextrines and British gums and explains the great flexibility and versatility of dextrines for a variety of uses. Yellow dextrines are the most highly converted of all the converted starches, and are relatively thin cooking. It is possible to prepare concentrated solutions of yellow dextrine of 50 to 60% solids that will remain stable with very little thickening for a period of months. Such solutions are very tacky and sticky. One would think that they would be of considerable interest for pigment coatings, but unfortunately they do not have the strength characteristics required for that use.

On the other hand, yellow dextrines do have a number of uses which are related to coating. Because concentrated solutions of yellow dextrines can be handled, and because they are readily soluble in cold water, they have found a considerable use in the manufacture of gummed papers. This is a coating type of operation and hence should be considered under the general heading of coating adhesives. The common gummed label frequently depends entirely upon a yellow dextrine for its remoistening adhesive. The backs of postage stamps or the front seal of envelopes are all yellow dextrine types.

DEXTRINES IN PIGMENT COATINGS

Returning to the use of dextrines in pigment coatings, which is our principal subject today, the converted starches which have been found of most use are oxidized starches, enzyme-converted starches, white dextrines, and, to some extent, the British gum types. In order to bring out the characteristics of the white dextrines and British gums particularly, it might be interesting to compare the viscosity characteristics of these two materials with those of typical oxidized starch types and enzyme-converted starch when cooked with water only. In all cases corn products were used. Figure 1 gives the viscosity characteristics of these types in paste form at a series of temperatures In determining these data the methods employed were as follows:

Slurries of commercial products with known degrees of conversion were prepared at 25% anhydrous starch solids, cooked at 190°F. for 15 min. and water loss replaced. Viscosities were measured on the Brookfield viscometer having four speeds 2, 4, 10, and 20 r.p.m. Cooling the cooked pastes was done under controlled agitation and time in a water bath to the temperature for testing. Temperatures of 150, 120, and 100°F. were selected to show the effects on the various converted starch products over the range encountered in coating applications. One hundred and fifty degrees Fahrenheit represents the lowest temperature at which the cooked paste is normally handled in the plant, and the lower temperature, 100°F., that can be expected in the finished color.

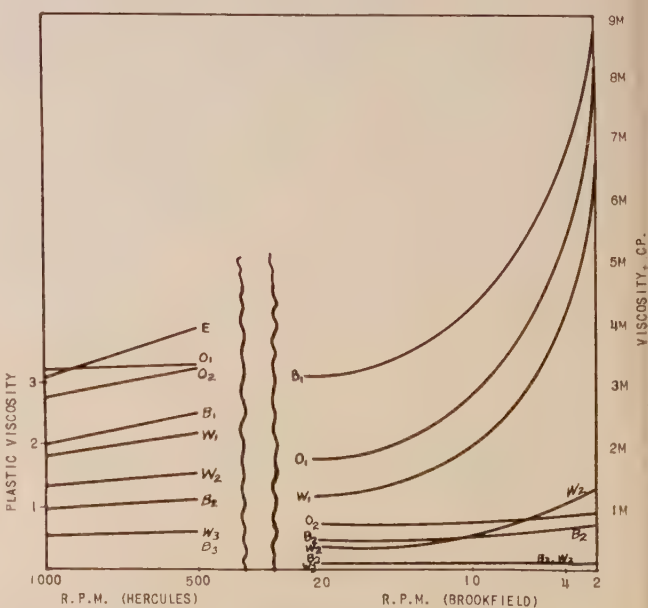

Fig. 2. Stability of starch cooks to agitation at 100°F.

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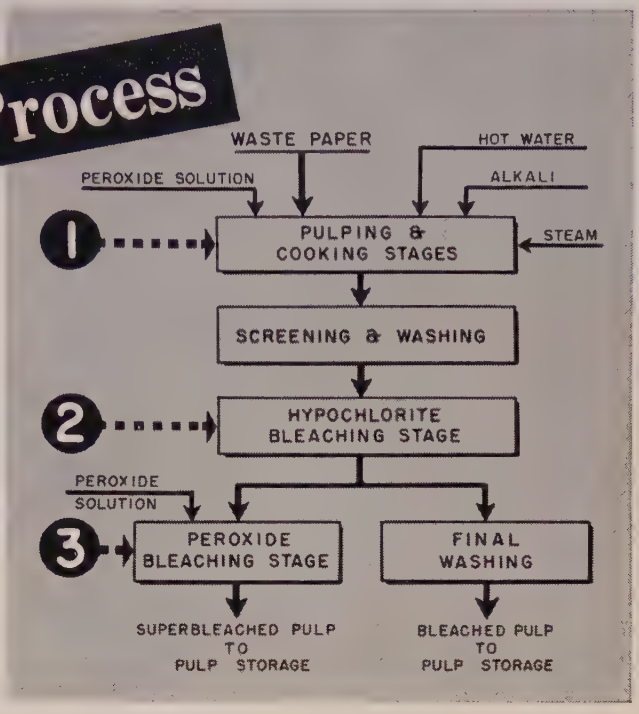

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In Fig. 1, and in all others, one enzyme conversion, designated E-2 is compared with two oxidized starches referred to as O-1 and O-2 and three British gums referred to as B-1, B-2, and B-3 as well as three types of white dextrines referred to as W-1, W-2, and W-3. In each case, as the number designation increases from 1 to 2 to 3 the extent of conversion increases for each type. While the comparison is not exact between types, different types with the same number designation, such as W-2 and O-2, are of the same order of magnitude in conversion. Products in the 2 and 3 range are normally used for off-machine coating.

In Fig. 1, the viscosity at 2 r.p.m. was selected in order to show the absolute viscosity, eliminating the effects of shear as much as possible. However, it should be noted that the same general shapes, or patterns, are still observed at other speeds. It also will be noted that as the viscosity is increased, or as the amount of conversion is decreased the tendency of all types of products is to thicken as the temperature of the paste is lowered. The British gums are more stable with respect to temperature and do not thicken as much on cooling as do the other types. White dextrines and enzyme-converted starches, on the other hand, are the most unstable in the lesser converted types and tend to thicken considerably on cooling. Oxidized starches are intermediate in stability. When these curves are compared, one should keep in mind that the unstable types thicken a good deal even before their pastes have been cooled to 150°F. The more stable types show their stability most in cooling from 130 to 100°F.

The effect of agitation is of extreme importance in clay coating. The effects as illustrated in Fig. 2 cannot be compared exactly with the behavior of coating color under agitation but there is a correlation between the two. A study of the dextrines themselves will give an indication as to the effect in the coating color. Small degrees of agitation are given in the right-hand portion of Fig. 2, and that data would be of principal interest in off-machine coating. The data of the left-hand portion of Fig. 2 illustrate the effect of more vigorous agitation and would be of more interest in machine coating, particularly when the data are considered also in the light of the effects of agitation under lower degrees of agitation.

Figure 2 demonstrates that in the lesser converted range, the white dextrines show a considerably greater reduction in viscosity than do equivalently converted British gums and

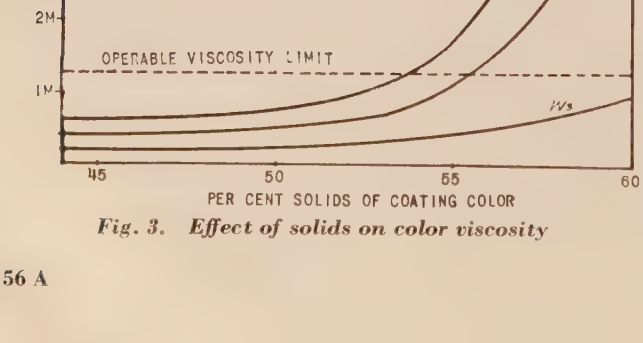

Fig. 3. Effect of solids on color viscosity

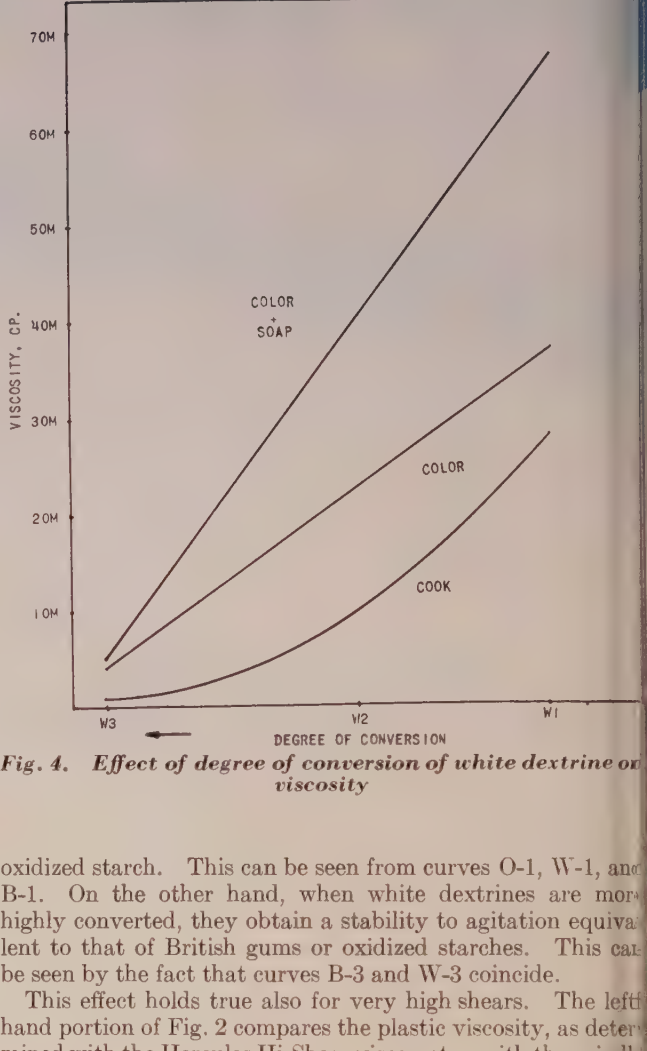

Fig. 4. Effect of degree of conversion of white dextrine on viscosity

oxidized starch. This can be seen from curves O-1, W-1, and B-1. On the other hand, when white dextrines are more highly converted, they obtain a stability to agitation equivalent to that of British gums or oxidized starches. This can be seen by the fact that curves B-3 and W-3 coincide.

This effect holds true also for very high shears. The left-hand portion of Fig. 2 compares the plastic viscosity, as determined with the Hercules Hi-Shear viscometer, with the spindle speeds in revolutions per minute. In the very high shear range the drop in plastic viscosity is at a greater rate with the lesser converted materials than with the more highly converted. This may be seen by comparing the slope of curve W-1 with W-3. Figure 2 also shows that while the viscosity of the enzyme conversion was too high to plot on the low shear scale, its viscosity is comparable to the lesser converted starches at high shear. This does not hold true exactly from type to type of conversion, but in general, it is true that as the degree of conversion is increased then the tendency to break down with agitation is decreased.

In off-machine coating an adhesive is preferred which has relatively good stability and low viscosity. A free-flowing color slip is easy to brush out into a smooth coating. Pasty types of products tend to thicken and give nonuniform results. Thus, stable-type dextrines and oxidized starches have been used for many years for off-machine coating. It is possible to obtain lower viscosities with white dextrines, and yet get good strengths, than can be done with oxidized starches or enzyme conversions. It is not possible to convert cornstarch by the oxidation process to get the low viscosities possible with white dextrines. The same thing is true to some extent with enzyme-converted starches. It is difficult to convert enzyme starches uniformly and maintain strength characteristics as compared to the more converted white dextrines.

The data presented in Fig. 3 were obtained by plotting color viscosity at 100°F. against the per cent solids of coating colors having 15% binder based on the pigment. The viscosity values in centipoise units were determined with the Brookfield viscometer at 20 r.p.m. using the no. 3 spindle. All colors were dispersed to their minimum viscosities. The dotted line across Fig. 3 represents the approximate viscosity

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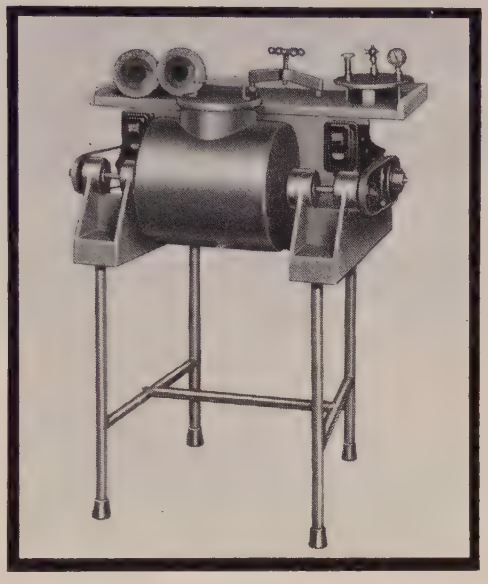

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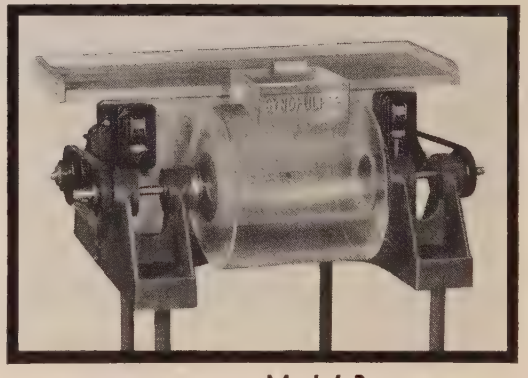

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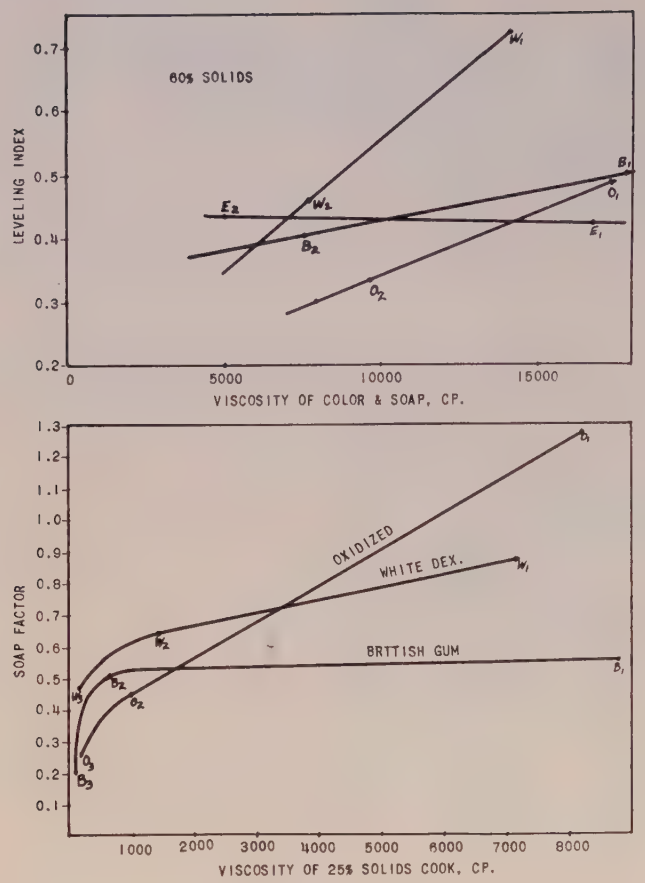

Fig. 5.

at which these colors could be successfully applied on most types of off-machine coaters. All of the materials plotted are used as off-machine coating binders, but O-2 and W-3 are the more commonly used. The strength characteristics of these materials are within the same order of magnitude. Oxidized starches offer advantages in high water retention which make them attractive for coating work at less than 50% solids. On the other hand, it can be readily seen that the more converted white dextrines are applicable in much higher solids ranges than would be possible with the oxidized starches.

Thus it is apparent that the more converted white dextrines offer good strength, adequate stability, high uniformity, and lower viscosity characteristics which permit good coating operations at considerably higher solids than is possible with other materials.

To evaluate the possibilities of converted products in regard to their adaption to roll coating, it was necessary to study several additional properties of these products. One of these investigated was the soap reactivity and its relation to leveling index.

Figure 4 illustrates the effect of soap on the color viscosity when white dextrines W-1, W-2, and W-3 were used. Actual viscosities of the dextrine-water cook, the color, and the color plus soap are plotted against the degree of conversion of the dextrine. It should be noted that the reactivity to soap increases considerably as the extent of conversion of the dextrine is decreased. A simplified way of recording this effect of soap on coating colors is to express it as the soap factor. This factor may be defined as the difference between the coating color viscosity, with and without soap, divided by the coating color viscosity without soap. Thus, this factor expresses the relative soap reactivity of any given degree of conversion, independent of color viscosity.

The soap factor was calculated for each dextrine from the data presented in Fig. 4. The factors so calculated appear as a curve for white dextrine in the lower portion of Fig. 5. Similar curves were determined in the same way for British gums and oxidized starches, and they also appear in the lower portion of Fig. 5. The viscosities of the starch cooks were determined at 25% solids with the Brookfield viscometer at 2 r.p.m. at 100°F. The soap (0.5% on pigment) used was a commercial flake commonly used for coating purposes. The coating color contained 60% total solids and had 15% binder on the pigment.

In Fig. 5 also, it can be seen that the British gums show no increase in soap reactivity with lesser degrees of conversion, nor do they show a satisfactory rate of increase in leveling index.

Both oxidized starches and white dextrines show high increases in soap reactivity with decreasing degrees of conversion. This is particularly true with oxidized starches, indicating that they might show very high leveling indexes. Contrary to this, the leveling index study as shown in the upper portion of Fig. 5 indicates that this is not necessarily so. Even though the soap reactivity rate of increase is high, the oxidized starches do not yield high leveling indexes. On the other hand, the white dextrines show very high leveling indexes, as well as a high rate of increase in leveling index with decreases in amount of conversion. In other words, sensitivity to soap is not directly related to the leveling index.

While it might be expected that reducing the extent of enzyme conversion would give increases in leveling index, our data show the opposite effect to be true. This may have some bearing on the difficulties which machine coaters have with their high viscosity enzyme conversions for lower solids work.

For roll coating, then, it will be noted that white dextrines: (1) offer high leveling indexes for all degrees of conversion, (2) give increasingly higher leveling indexes with minimum reduction in the extent of conversion, and (3) give higher viscosities at point of application of color to paper with lower initial color viscosities than is possible with enzyme conversions. While there are certain drawbacks to present white dextrines, the white dextrine type of conversion may offer properties of special interest for machine coating.

In this paper we have attempted to review the general characteristics for the different types of dextrines and their properties with particular emphasis on their use in pigment coatings. In order to bring these facts out more clearly, the dextrines have been compared with the oxidized starches and enzyme-converted starch commonly used for this type coating. Commercial cornstarch products have been used in each case. In general, the white corn dextrines offer characteristics of interest to both off-machine and machine coaters. Higher strengths for lower viscosities can be obtained in this way than with any other type of conversion. The dextrine method of conversion offers a degree of control and versatility which is not as easily obtained with the other types of conversions.

Oxidized Starch for the Paper Industry

J. M. NEWTON

THE terms "oxidized starch" and "chlorinated starch" are used interchangeably in the paper industry to describe one type of thin boiling modified starch. The term "chlorinated" is probably applied to this type of starch because the most common agent used commercially for starch oxidation is sodium hypochlorite. Chlorinated is a misnomer because the chlorine does not substitute into the starch molecule as the name infers, but rather serves to provide the oxidizing potential. This oxidation is brought about by the reduction of the positive valent chlorine in NaCl with the subsequent release of $^1/_2$ mole of oxygen. A further reason for using the

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term oxidation is the possible preparation of oxidized starches by nonchlorine oxidants, such as peroxides, permanganate, Activin or Chloramin-T, perborates, persulphates, hexavalent chromium salts, and other oxidizing agents.

From a chemical standpoint oxidation of starch with halogens appears to give four different types of reactions: (1) oxidation of aldehyde groups to carboxyl groups, (2) oxidation of primary alcohol groups to carboxyl groups, (3) oxidation of secondary alcohol groups to ketone groups, and (4) oxidation of glycol groups to dialdehydes and then to dicarboxylic acids.

The mechanism for the oxidation of starch with hypochlorite under commercial conditions is not known, but it is possible that any or all of these types of oxidation may occur simultaneously. The hypochlorites lack the specificity required for any one type of oxidation in such a polyfunctional molecule as the starch molecule. Such specificity of oxidation does occur, however, if the starch molecule is oxidized with periodate. The classical work in the field of periodate oxidation of starch is beyond the scope of this paper, but anyone interested should review the work of Hixon, Caldwell, Hudson, Purves, and others. Here, however, oxidation is carried far beyond that used in commercial oxidations, periodate oxidation giving one or more equivalents of oxidation per anhydroglucose unit while commercial oxidations give one equivalent of oxidation for 25 to 100 anhydroglucose units.

Although the exact mechanism is not established, commercial oxidation definitely introduces carboxyl groups into the starch molecule, the finished product having some of the characteristics of an organic acid. Since excess caustic is present during oxidation the acid groups are neutralized and occur as sodium salts. The proportion of free carboxyl groups occurring in the finished product is dependent upon the finished pH. The average oxidized starch has approximately 90% of the carboxyl groups neutralized with sodium.

Recently, Pollock and Campbell reported that considerable oxidation occurs at the glycol group. This gives rise to free formic acid during oxidation and results in a break in the carbon chain between the third and fourth carbon atom. Establishment of this observation would aid in explaining some of the viscosity changes caused by oxidation.

COMMERCIAL OXIDATION METHODS

Usually, aqueous starch suspensions (30 to 34% dry substance starch) are treated with a hypochlorite solution (6 to 9% chlorine) containing a slight excess of sodium hydroxide. The decomposition of the hypochlorite and the oxidation of the starch by the nascent oxygen are both exothermic reactions.

The starch solutions increase in temperature unless adequate cooling is provided. After several hours, when the reaction is judged to be approaching completion, a sample of the starch is removed, filtered, washed, and the fluidity or viscosity of a paste prepared from the cake is determined. When the proper degree of oxidation is reached the oxidation is stopped by adding sodium bisulphite, the slurry is adjusted to the desired pH, filtered, washed, and dried. By varying the quantity of hypochlorite added and the temperature, pH, and time of the reaction, the full range of oxidized starches is produced.

PROPERTIES OF OXIDIZED STARCH

The finished product retains the granule structure, is insoluble in cold water, shows typical starch-iodine reactions, retains the typical polarization crosses, and has the same general appearance as unmodified starch. It differs in that it is whiter in color and the granules have definite surface fissures which are evident when carefully observed under the microscope. In contrast to the slight alteration in granule appearance the physical properties of the solutions are markedly altered. When gelatinized in water unmodified starch yields

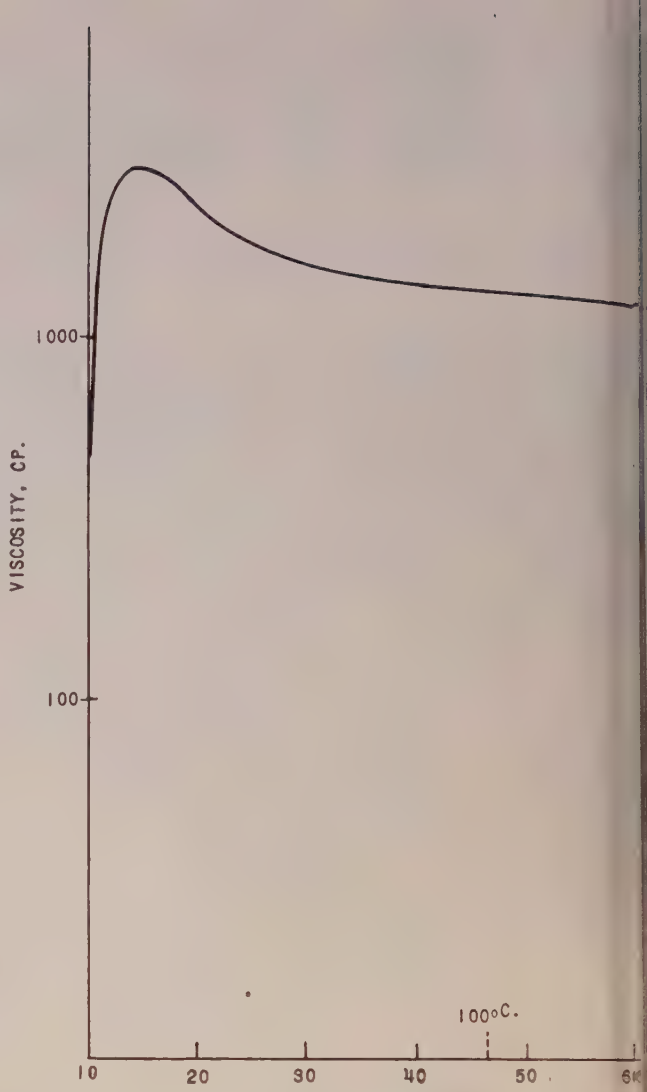

Fig. 1. Cooking curve for 10% unmodified cornstarch

heavy pastes, while the oxidized starches at the same concentrations give thinner bodied solutions, the degree of thinness being dependent upon the extent of the oxidation. The oxidized starches have a lower rate and less tendency to gel when cooled. Films formed by drying oxidized starch are tough and horny in comparison with the brittle, pliable films of unmodified starch.

The fluidity classification utilized by manufacturers to characterize oxidized starch has always been confusing to the user. Fluidity refers to the volume of a cooked paste of specified concentration which will flow through a standardized orifice in a specified period of time as compared to a specific volume of water which flowed through the same orifice in the same length of time. For example, a "60 fluidity" starch means that 60 ml. of the starch suspension have flowed through the standard orifice in the same period of time required by 100 ml. of water. The concentration of the paste is adjusted so that the values range from 0 to 100. The larger the number the greater the degree of modification. The most highly oxidized products are not classified by a numerical system, but are referred to as starch gums. The term "gum" probably originated from the fact that the relatively clear solutions prepared from the highly oxidized starches resemble vegetable gums in appearance and properties.

In order to show the marked modification that occurs in the viscosity characteristics of sodium hypochlorite oxidized starches we have prepared a series of starch pastes under standard conditions and observed the viscosity throughout the cooking cycle. The starch pastes were prepared by suspending the dry starch in water at room temperature and then cooking these suspensions in the Corn Industries Vis

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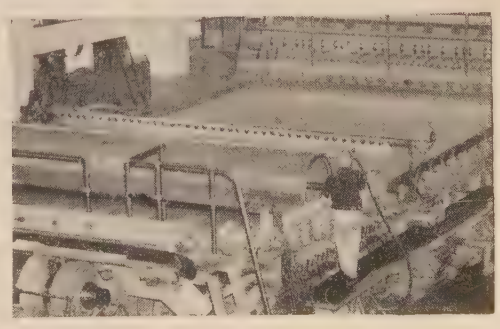

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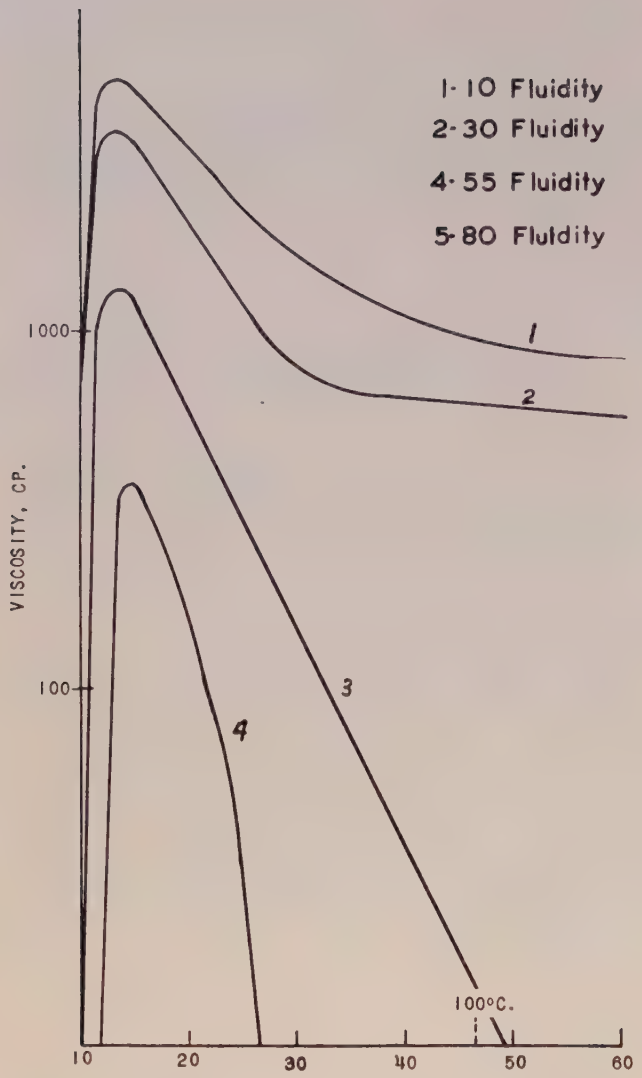

Fig. 2. Cooking curves for low oxidized cornstarches

cometer. Constant agitation is applied throughout the cooking cycle. The temperature increase follows the same pattern in all cases.

Figure 1 shows the viscosity curve for unmodified starch at a concentration of 10% dry solids. The viscosity increases to a maximum at 14 min. at a temperature of 76°C. The viscosity decreases at a rapid rate for the next 15 min. and then becomes relatively constant.

Figure 2 shows similar data for commercially oxidized starches under comparable conditions except that the concentration is increased to 12%. At this concentration common starch would be beyond the 10,000 cp. maximum. The four starches shown would be classified as 10, 30, 55, and 80 fluidity. The 10 and 30 fluidity starches are low fluidity oxidized starches. Their viscosity curves have a marked resemblance to common starch. The 55 and 80 fluidity oxidized products are characterized by a marked reduction in their viscosity with increasing temperature. Apparently, sufficient chemical modification has occurred in these products to allow for a rapid disintegration of the granule and perhaps the molecule under these cooking conditions. The pastes from these intermediate oxidized starches vary markedly from common starch in their appearance and properties, exhibiting the shorter cooking time, greater clarity, higher fluidity, lower rate of congealing, and softer gel characteristicn of oxidized starch.

Figure 3 presents two typical commercial oxidized starch gums at 30% concentrations. The 80 fluidity product from Fig. 2 has been included as curve no. 1 to compare the viscosity of an intermediate oxidized starch with the gum starches. Here again the sharp drop in viscosity is observed, and th typical leveling off in viscosity is shown with gum no. 2.

The maximum temperature reached in all cases was 100°C at 47 to 48 min. If boiling temperatures are reached in shorter time the minimum viscosities are reached more quickly

These figures strikingly present the marked alteration i the viscosity characteristics of starch introduced by oxida tion. At three times the concentration the gum starches ar thinner than common starches. Actually, 7 parts of the lo viscosity gum give a viscosity comparable to 1 part of com mon starch when prepared under comparable conditions.

USE OF OXIDIZED STARCH IN CLAY COATINGS

From an adhesive standpoint unmodified cornstarch woul give the paper coater the maximum amount of adhesive pe dollar. However, as discussed previously, unmodifie starches give pastes with such high viscosities that they can not be used in coating work. Further demands for re duced viscosity are imposed by the high solids coatings re quired for machine coating operations.

In order to meet the viscosity requirements of the coate starches must be highly oxidized. Only the gum type oxi dized starches meet this requirement.

The particular gum to use for an individual mill depend upon the coating process used at the mill and on the mill con ditions which will be encountered in this use. The type o pigment used, the condition of the mill water, the cooking equipment, the coating process, and the base stock being use

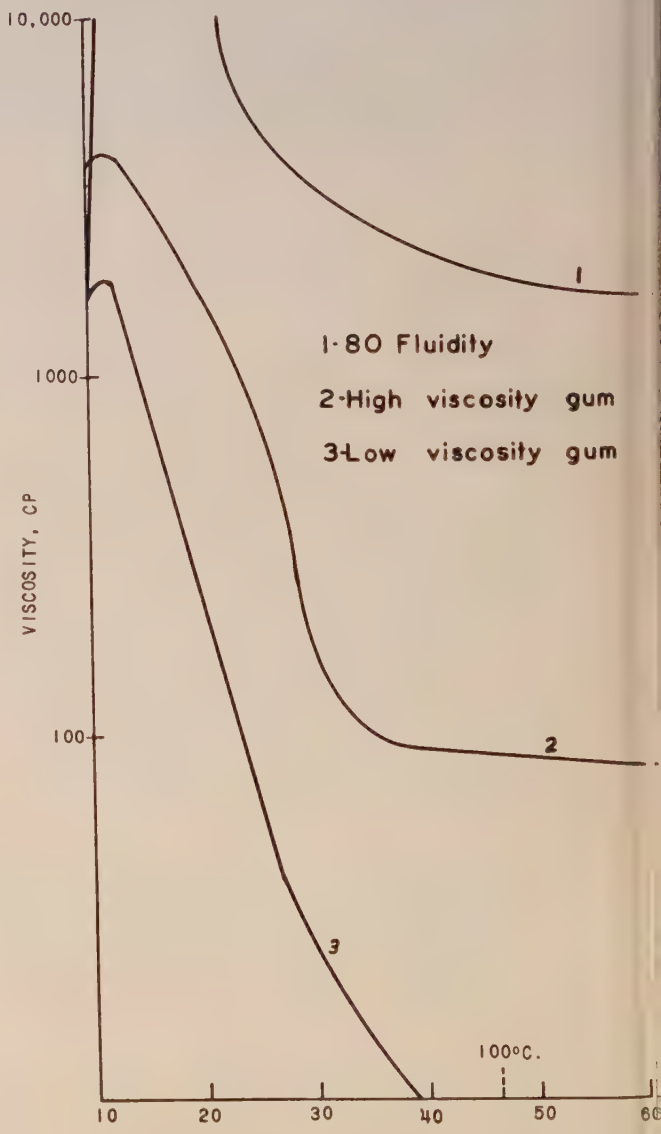

Fig. 3. Cooking curves for high oxidized cornstarches

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influence this decision. Each coating mill must be treated as an individual problem and the decision, as to the correct gum to use, must be made after a careful study of the controlling factors.

The correct procedure to use in preparing coating colors, with oxidized starch gums as the adhesive, is also dependent upon individual mill conditions. Generally, 3 parts cool water and 1 part gum is agitated to form a smooth slurry. The quantity of gum used varies from 13 to 20% based on the total clay to be used in the color. Less water is undesirable from a standpoint of gelatinization, but more water is advantageous if it can be tolerated and still maintain the solids content in the finished color. The temperature of the slurry is raised rapidly with good agitation to 200–210°F. and held at this level approximately 15 min. The starch is then cooled rapidly to 150°F. The temperature after cooling will vary, but as high a temperature as possible is desirable since the starch paste remains more fluid at higher temperatures and mixes more readily with the clay slip. The cooled paste is then added to the clay slip, mixed thoroughly, and diluted to the optimum solids content for best operations.

All users of starch adhesives should remember that starch pastes are relatively labile, colloidal systems. The curves shown previously demonstrate the marked viscosity changes resulting from prolonged cooking. Overcooking is to be avoided. Excessive high-speed agitation and excessive circulation especially with centrifugal pumps may also thin and weaken coating colors. All such degradations may cause loss of adhesive strength and result in reduction in printing qualities in the finished sheet.

Viscosity reduction in starch pastes is necessary if they are to be used as coating adhesives. In addition, we must recognize that the greater the degree of conversion, the less adhesive strength per pound of starch. Accepting this general fact, the starch chemist has been exerting efforts to improve oxidized starches by giving them improved flow properties with least reduction in average molecular size. In true solutions, molecular size and viscosity are directly correlated, and such a problem would not exist. However, in the complex starch colloidal system where viscosity is dependent on the swollen granules and molecular agglomerates as well as molecular dispersions, the problem actually exists and invites investigation. Improved oxidized starches can result from such investigations.

ECONOMIC CONSIDERATION

The excellent quality of the coated paper prepared with oxidized starch as the adhesive, the convenience of the operation, the freedom from responsibility and cost of controlling the conversion, and the uniformity of the starting material justify wide use as a coating adhesive. Oxidized starches are premium priced products and in many cases cannot be justified on an economic basis. The gum starches used for coating usually carry a premium of about $1.75 per 100 lb. over pearl cornstarch.

The premium on gum starches is necessitated by: (1) chemical costs (principally chlorine and caustic soda), (2) high solubilization losses in processing, (3) increased manufacturing costs (principally reduced drier capacity), and (4) increased laboratory control costs.

The successful paper coater will analyze his production costs and weigh the advantages of the oxidized starches very carefully. Many coaters are able to justify the oxidized products, particularly for specialty uses.

CONCLUSION

Oxidized cornstarches are carefully controlled modified thin boiling starches which have excellent color, desirable working properties, good adhesive power, and uniform viscosities. These properties when utilized by the paper coater assist the coater in producing the highest quality products that will consistently satisfy the consumer of coated paper.

Use of the Corn Industry Viscometer for the Evaluation of Starches and Enzymes

M. J. MASON and R. J. HORST

In the past numerous instruments have been designed and manufactured for the purpose of measuring the viscosity and flow properties of starches and other colloid materials. However, only a few of these instruments have been able to indicate or measure continuously the consistency changes that occur when starch suspensions are subjected to heat, chemical treatment, and enzyme degradation. The limitations of these few instruments were apparent to most of the starch manufacturers and starch consumers. However, with the adoption of the Corn Industries Viscometer by the wet milling industry the evaluation of starch and starch products is becoming more simplified and standardized.

As Kesler and Bechtel (*1*) have already pointed out, the viscosity and pasting characteristics of modified and unmodified starch can be obtained with ease and accurate duplication of results. Later Bechtel and Kesler (*2*) demonstrated that the instrument could be used to follow the liquefaction of cornstarch with enzymes.

Cornstarch is converted with enzymes within a rather wide range of solids content in order to fulfill the particular requirements of both the adhesive and paper industries. This range may vary from 10 to 45% starch solids and it is beneficial to determine beforehand the converting characteristic of the starch selected, the approximate amount of enzyme to use, the proper converting schedule to employ, and to obtain the viscosity value after the conversion is completed.

Converting schedules using the Corn Industry Viscometer have been worked out whereby the converting characteristic of starch at 20% solids and at 40% solids can be followed continuously throughout the heating and cooling cycle. Procedures for carrying out the different schedules are as follows.

After preheating the water bath of the viscometer to 45°C., a starch slurry weighing 1000 grams and containing 20% solids is adjusted to pH of 6.5 to 7.0. The slurry at room temperature is poured into the viscometer cup and the water bath heater turned on. Thirty-seven and a half milliliters of 1.60% solution of enzyme are added when the starch slurry reaches 45°C. (*Note:* For accuracy and duplication of results it is necessary to weigh 4.00 grams of enzyme and make up to a volume of 250 ml. with distilled water. Only fresh solutions of the diluted enzyme should be used and any remaining solution should be discarded after 8 to 12 hr.) When the temperature of the paste reaches 71.0°C., then the converting schedule given in Table I is followed by manual operation of the thermostat.

The reproduced chart shown in Fig. 1 illustrates the continuous recording of the viscosity changes that occur when the starch paste is being liquefied by the enzyme. After the conversion cycle is completed and the paste cooled to 32°C., the adhesive is removed from the viscometer cup and allowed to remain undisturbed for 24 hr. Then the material

M. J. Mason, Section Director, and R. J. Horst, Research Chemist, Central Research Dept., Anheuser-Busch, Inc., St. Louis, Mo.

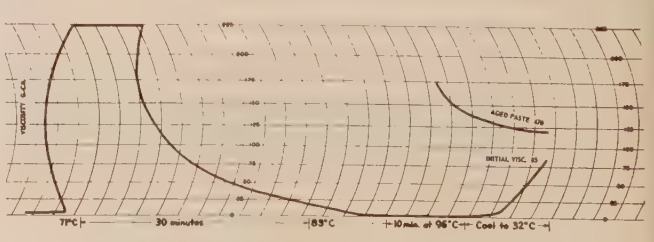

Fig. 1. Continuous converting schedule using 20% starch solids and 0.3% enzyme

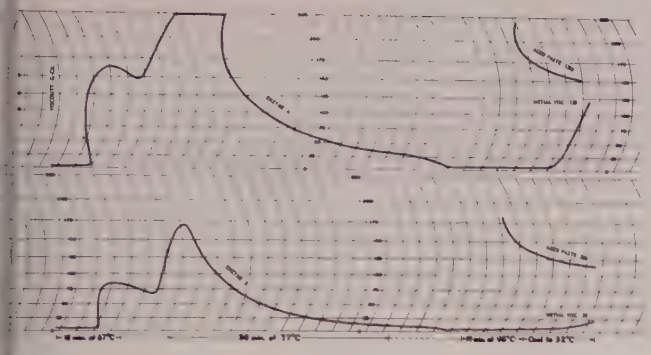

Fig. 2. Conversion characteristics of two different enzymes using a two-stage converting schedule and 0.2% enzyme

is poured back into the viscometer and another viscosity determined at 32°C. by allowing the agitator to run for 16½ min. before a reading is taken. The increase in viscosity of the converted starch after aging 24 hr. is an indication of the "set-back" quality of the starch.

Since various conversion procedures are used in coating preparations, another common type of converting schedule has been tried whereby the temperature and the time are held constant for two periods during the converting range. Table II indicates the cooking schedule to be followed for this type of conversion. This procedure was found to be convenient for comparing the relative liquefying rates of two different enzymes and the curves shown in Fig. 2 illustrate the differences in the converting power of the enzyme selected for this test. As shown, a smaller amount of enzyme (25 ml. of a 1.60% solution) can be used for the two-stage converting schedule than in the case where a continuous converting schedule is employed. This is primarily due to the fact that the enzyme is given a longer period of time to act on the starch paste while in the converting range. In respect to the relative activity of the two enzymes, it can be seen that enzyme B liquefies the starch to a greater extent than enzyme A. The differences between the initial viscosities at the end of each converting cycle are significant while the viscosities of the aged pastes show quite a substantial difference.

Often conversions are carried out at very high starch concentrations, and great care should be exercised in selecting a converting schedule that will result in the proper liquefaction of the starch. The cooking schedule shown in Table III is used when starch is converted at 40% solids or higher. Figure 3 illustrates the type of curves obtained when modified and unmodified starches are converted using a four-stage converting schedule. Although the illustration does not show the inactivation and cooling periods of the complete

Table I. Time and Temperature Control for a Continuous Converting Schedule Using 20% Starch Solids and 0.3% Enzyme

Time control, min.	*Temperature of bath setting, °C.*	*Temperature of paste, °C.*
0	78.5	71.0
3	78.5	73.0
6	78.3	75.0
9	79.0	76.0
12	79.2	77.0
15	80.2	78.0
18	81.0	78.8
21	81.5	79.2
24	82.0	80.0
27	85.0	81.0
30	88.0	82.6
Raise in 12–13	98.0	. . .
Hold for 10	98.0	95–96
Cool for 14–16	15–25	32.0

Table II. Time and Temperature Control for a Two-Stage Converting Schedule Using 20% Starch Solids and 0.2% Enzyme

Time control, min.	*Temperature of bath setting, °C.*	*Temperature of of paste, °C.*
Hold for 10	70.0	66–68
Raise in 8–9	80.0	. . .
Hold for 30	80.0	76–78
Raise in 14	98.0	. . .
Hold for 10	98.0	95–96
Cool for 14–16	15–25	32.0

cycle, it is interesting to observe that the viscoisty changes are quite different during the conversion period and it is obvious that the modified starch converts with greater ease than the unmodified starch. It is significant to point out that starches converted at 40% solids require 0.75% enzyme based on the weight of the starch while conversion with 20% starch solids requires 0.2 to 0.3% enzyme.

The three converting schedules just described were made using single strength enzyme preparations commonly supplied by the enzyme manufacturers for laboratory and pilot-plant evaluation tests. Before any conversions are attempted, it is important to know whether the enzyme selected is single

Table III. Time and Temperature Control for a Four-Stage Converting Schedule Using 40% Starch Solids and 0.75% Enzyme

Time control, min.	*Temperature of bath setting, °C.*	*Temperature of of paste, °C.*
Hold for 15	71.0	68–69
Raise in 2½	75.0	. . .
Hold for 15	75.0	71–73
Raise in 2½	78.0	. . .
Hold for 15	78.0	74–76
Raise in 6–7	81.0	. . .
Hold for 30	81.0	77–79
Raise in 12–13	98.0	. . .
Hold for 10	98.0	95–96
Cool for 14–16	15–25	32.0

strength or the multiple strength type often used in factory conversions. When studying different enzymes, it may be necessary to increase or decrease the amount used in order that the viscosity value of the converted paste after cooling to 32°C., reads between 25 and 225 G.-cm. If this procedure is followed, the viscosity reading of the 24-hr. aged paste will rarely exceed the instrument's maximum load of 1800 G.-cm.

INACTIVATION OF THE ENZYME

Two methods are commonly used to inactivate the enzyme after the liquefaction of the starch has progressed to the proper point. One method, and the one most commonly used, employs the use of heat while the other method depends on the addition of chemicals that are toxic to the en-

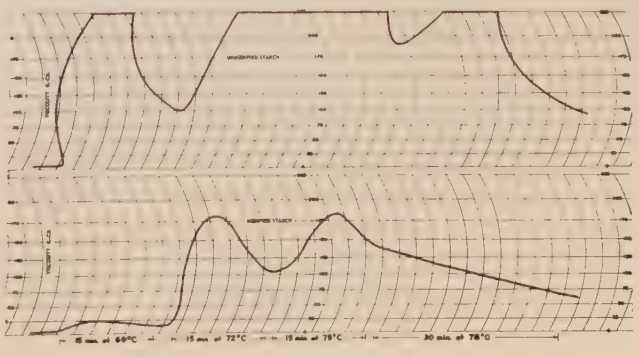

Fig. 3. Use of a four-stage converting schedule to compare different starches cooked at 40% solids and using 0.75% enzyme

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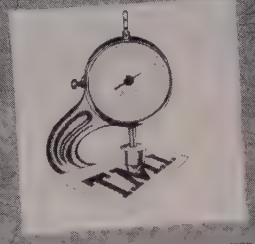

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Table IV. Differences in the Final Paste Viscosities Due to Variation of the Inactivation Time

Time held at 96°C., min.	*Viscosity after converting and cooling, g.-cm.*
10	800
20	560
30	332

zyme. A study was made on the effect of varying the heating time at the inactivation temperature of 95 to 96°C. Table IV shows the different viscosities obtained when the same starch sample was converted three times in the same manner, except that the heating time was varied from 10 to 30 min. The successive reduction of the viscosity can probably be attributed to the fact that a portion of the starch paste is underconverted and that heat and agitation contribute to the mechanical breakdown of the paste.

The use of the Corn Industries Viscometer to study the conversion of cornstarches with enzymes, indicates that this instrument is very adaptable to various types of converting schedules commonly used in factory practice. Conversions can be carried out on starches cooked at both low and high solids concentrations with good reproducibility of results. The converting characteristics of both modified and unmodified starches can be detected thus enabling the starch consumer to adjust for the differences before factory conversions are made. Also the liquefying action of various enzymes can be followed by continuous recording of the viscosity fluctuations throughout the conversion cycle.

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2. Bechtel, W. G., and Kesler, C. C., *Paper Trade J.* 125, No. 16: 35–39 (Oct. 16, 1947).

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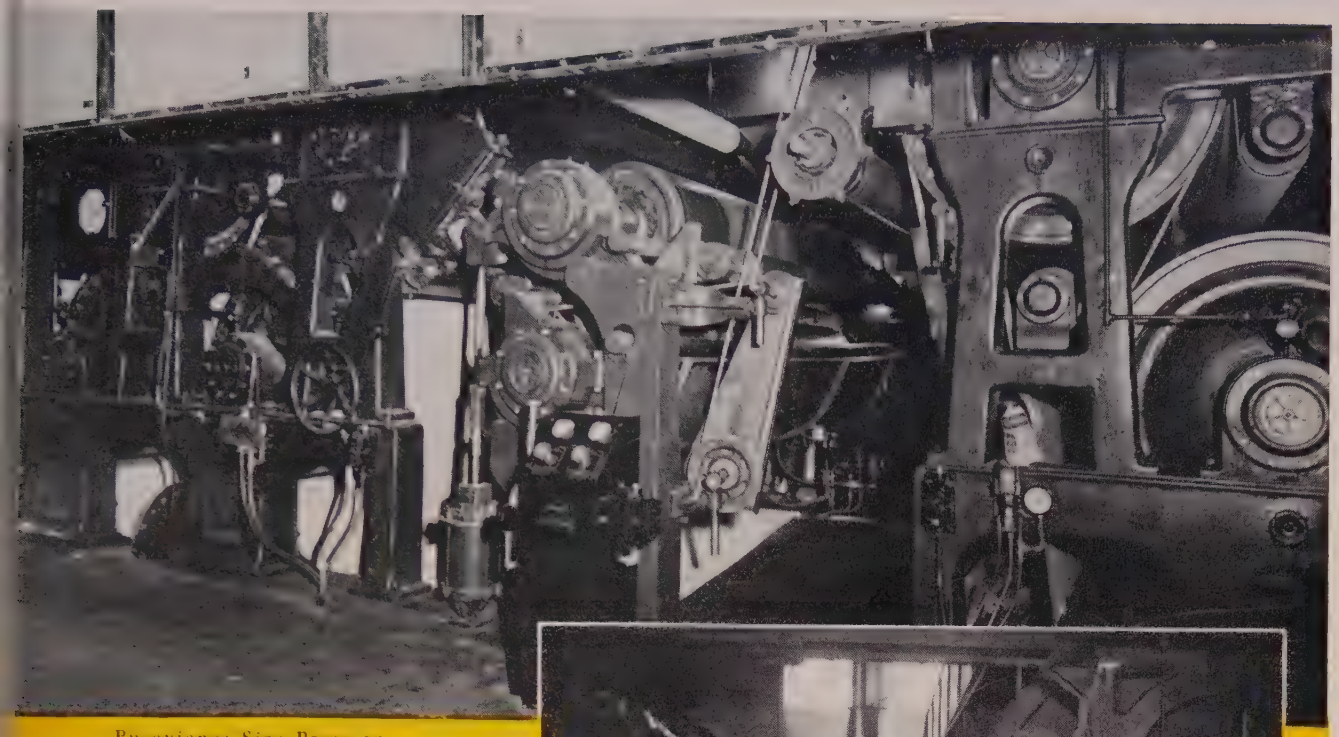

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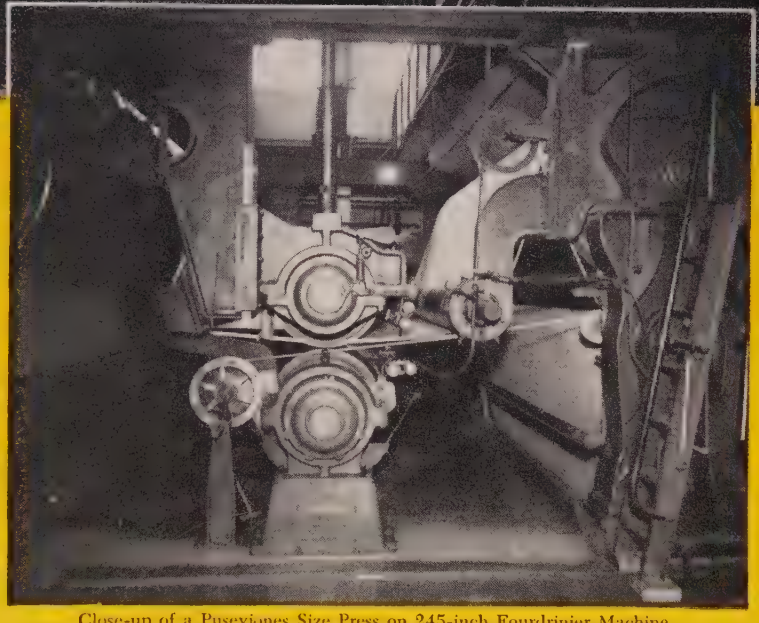

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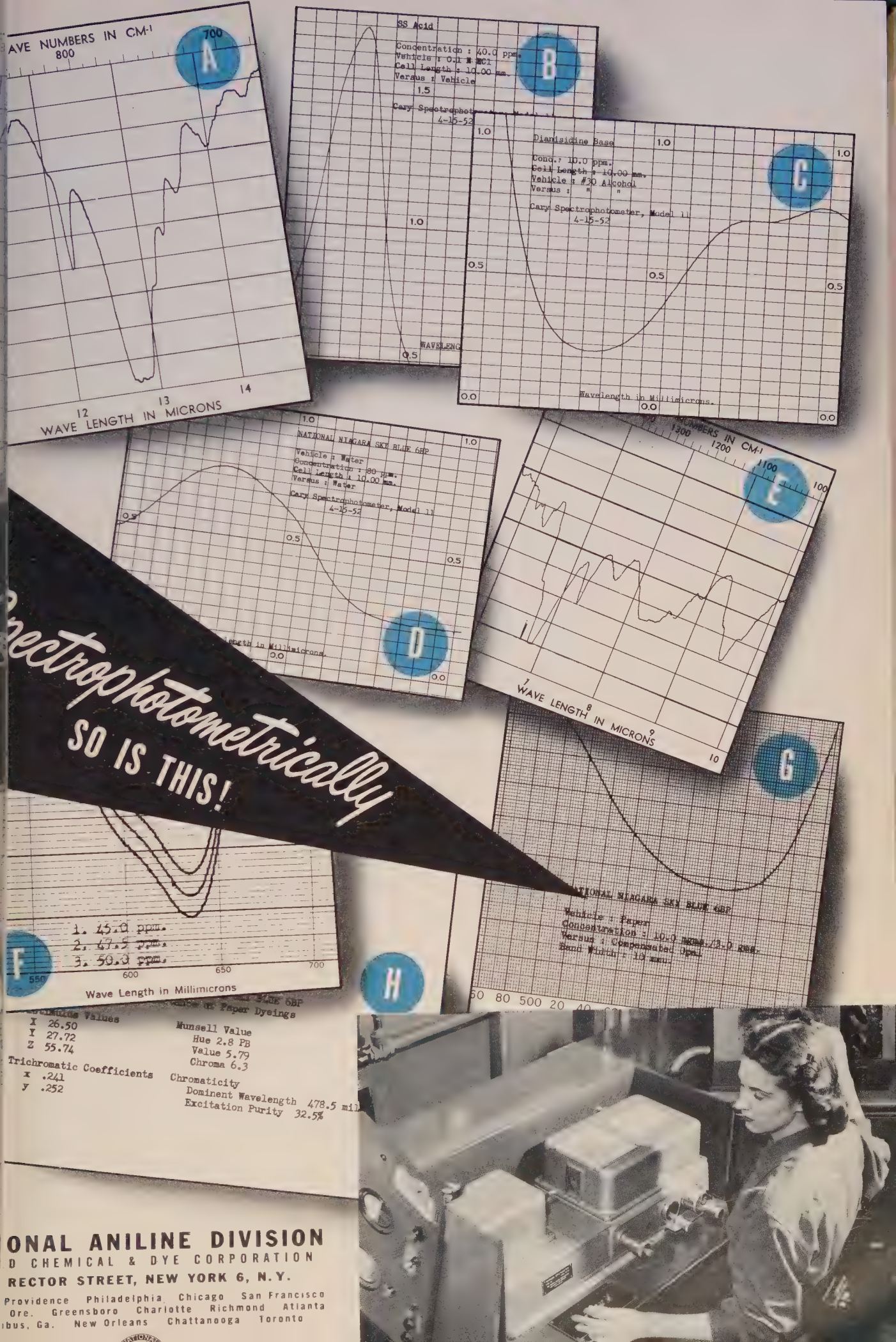
AVE NUMBERS IN CM-1
800
700
A
12
13
14
WAVE LENGTH IN MICRONS
SS Acid
Concentration : 40.0 ppm.
Vehicle : 0.1 N HCl
Cell Length : 10.00 mm.
Versus : Vehicle
1.5
1.0
0.5
B
Dianisidine Base
Conc.: 10.0 ppm.
Cell Length : 10.00 mm.
Vehicle : #30 Alcohol
Versus : " "
Cary Spectrophotometer, Model 11
4-15-52
1.0
0.5
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C
Wavelength in Millimicrons.
NATIONAL NIAGARA SKY BLUE 6BP
Vehicle : Water
Concentration : 80 ppm.
Cell Length : 10.00 mm.
Versus : Water
Cary Spectrophotometer, Model 11
4-15-52
D
Spectrophotometrically
SO IS THIS!
WAVE NUMBERS IN CM-1
1300
1200
1100
100
E
WAVE LENGTH IN MICRONS
1. 45.0 ppm.
2. 47.5 ppm.
3. 50.0 ppm.
F
550
600
650
700
Wave Length in Millimicrons
G
NATIONAL NIAGARA SKY BLUE 6BP
Vehicle : Paper
Concentration : 10.0 mgms./3.0 gms.
Versus : Compensated Opal
Band Width : 10 mms.
H
Tristimulus Values
X 26.50
Y 27.72
Z 55.74
Trichromatic Coefficients
x .241
y .252
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Hue 2.8 PB
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Manufacture of Paper from Glass Fibers

RTIN J. O'LEARY, BOURDON W. SCRIBNER, JOSHUA K. MISSIMER, and JAMES J. ERVING

aper made entirely of glass fibers, with no additive, developed at the National Bureau of Standards in operation with the Naval Research Laboratory for use n air filter. The paper may be useful also for filtration iquids, and should have extensive applications in elec- ic and other electrical equipment because of its ex- ent insulating properties and very high resistance to t. Certain modifications of the practice in making er from cellulose fibers were found necessary. The meter and length of the fibers have an important bear- on both strength and good formation. The best results e obtained with fibers having a diameter of 0.5 to 0.75 . A combination of long and short fibers, free from s, gives the strongest paper.

THE production of papers from glass fibers was ertaken in the experimental paper mill of the Na- al Bureau of Standards in cooperation with the val Research Laboratory as a part of that labora- y's program of work on the development of papers military uses. The need was for papers having such el characteristics, compared with cellulosic papers, a high degree of resistance to heat, chemicals, and dew; low moisture absorption; dimensional sta- ty; high permeability to air and water with very high ention of solid particles, desired for filters; electrical aracteristics associated with glass.

The suitability of glass fibers for such purposes has n recognized by others, and papers have been pro- ed from glass fibers with an admixture of vegetable synthetic fibers. It appeared, however, that an all- ss product would be decidedly superior to these pers for the uses contemplated. Accordingly, the rk to be described was directed toward the produc- n of all-glass fiber paper.

This report deals primarily with the production of per suitable for air filtration, but glass paper has been nd satisfactory for other uses. Paper of greater ength can probably be produced by the use of binding nts, but this has not so far been investigated.

The manufacturing process developed at the Bureau s been successfully applied to commercial manufac- e of the paper. Paper manufacturers were assisted production of it by demonstrations of the process in Bureau mill and, in some instances, by advisory pervision of its application in commercial mills by member of the Bureau staff.

The semicommercial papermaking equipment at the tional Bureau of Standards is adapted to the experi- ental manufacture of papers under conditions that ulate those of typical industrial plants. The re- lts obtained are indicative of what can be produced a commercial scale.

The equipment used in this particular work was a 50-lb. beater with copper-lined tub and manganese-bronze bars and plate, 29-in. fourdriner papermaking machine with a wire 33 ft. long, one press, nine 15-in. driers, and a reel. Handsheets were made with a sheet mold, a hydraulic press, and a sheet drier.

Detailed descriptions and photographs of the papermaking equipment of the Bureau are contained in a previous publication (*1*).

RTIN J. O'LEARY, JOSHUA K. MISSIMER, and JAMES J. ERVING, members he staff, and BOURDON W. SCRIBNER, Late Chief, Paper Section, National eau of Standards, Washington, D. C.

RAW MATERIAL

Four grades of "Fiberglas," graded according to diameter, were used: AAA, AA, A, and B fibers. These fibers were supplied by the Owens-Corning Fiberglas Co. Glass Fibers, Inc., has also supplied fibers that have been used successfully.

The diameter of the AAA fiber ranged from 0.5 to 0.75 mu. It feels like silk when dry and has a greasy feel when wet. The diameter of the AA ranged from 0.75 to 1.5 mu. This fiber is soft and feels like rayon when dry.

The A fiber ranged from 1.5 to 2.5 mu in diameter. There was a considerable amount of small particles of glass mixed in with the fibers. One should wear gloves when handling it. The fiber feels granular when it is wet. The B fiber ranges from 2.5 to 3.75 mu in diameter. This fiber is otherwise similar in characteristics to the A fiber.

The fibers are clean and can be furnished directly to the beater.

MANUFACTURING PROCEDURE

The manufacturing procedure using glass fibers had to be changed in several important details from the familiar procedures employed for cellulose fibers. These changes were necessitated chiefly by two significant differences in the properties of glass fibers as compared with cellulose fibers: first, that the glass fibers are much more brittle, so that they are easily broken by the usual papermaking technique; and second, that the glass fibers cannot be fibrillated and gelatinized like vegetable fibers.

Because of these properties, prolonged beating is not only unnecessary, but causes excessive breakage of the fibers. The glass fibers were added to the water in the beater in the usual way, except for some additional care to break up large masses of fiber as they were introduced. It was found that concentrations over 1% were undesirable, for at these concentrations undesirable breakage of the fibers occurred, and above 2% the stock became so bulky that it would not circulate properly. The settings of the beater roll were varied from 0.08 to 0.008 in. off the base plate. When the roll was lowered to the base plate the resultant paper contained numerous pinholes, a condition leading to poor filtering efficiency. Beating was continued only long enough to open up and separate the fibers and give

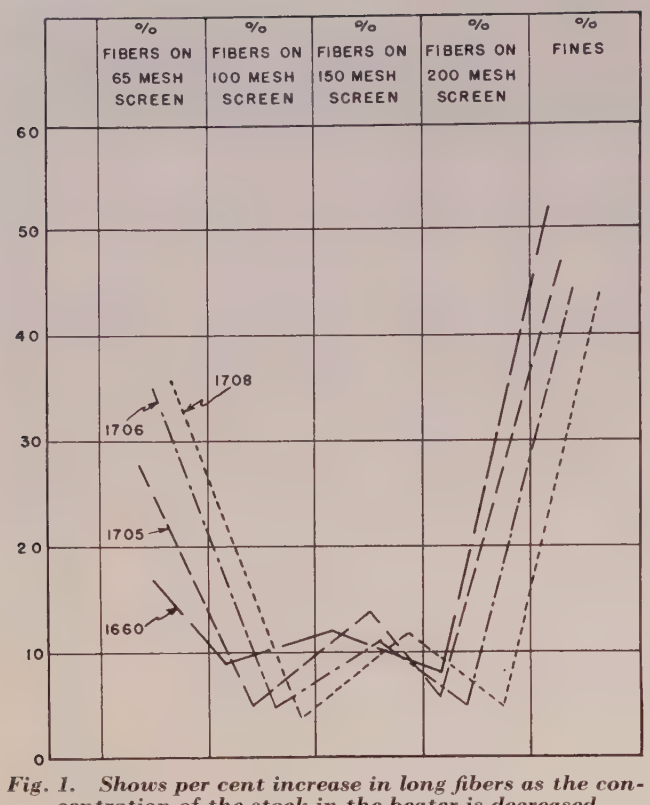


Fig. 1. Shows per cent increase in long fibers as the concentration of the stock in the beater is decreased

—— 1660, 2% conc.; — — 1705, 1½% conc.; —·— 1706, 1% conc. – – – – 1708, 0.7% conc.

a satisfactory suspension; the time required varied from 15 to 90 min., depending upon the concentration, roll setting, and grade of fiber. Longer beating resulted in considerable breaking of the fibers. The temperature of the stock in the beater was maintained at 72 ± 2°F. in all the runs.

The beaten stock was dropped to the chest and pumped in a continuous stream through the stuff box and into the fan pump, where it was mixed with the water drained from the paper machine.

The jordan was by-passed because tests made with a Bauer-McNett pulp classifier showed that when the glass fiber pulp passed through the fan pump, the long fibers were shortened by as much as 25% (Table I). This fiber breakage would result in a weaker paper.

The stock was propelled from the fan pump into the riffle box. As it flowed over the baffle boards in this box, heavy particles of glass in the stock, which would have caused irregularities in the finished sheet, settled to the bottom of the box. From the riffle box the stock by-passed the screens, going directly into the screen hopper and thence to the headbox and onto the paper machine wire. This by-passing of the screens was required because the glass fibers separate from the water and remain on the screen plates, unlike cellulose fibers which will pass through the openings in the screen

Table I. Loss in Long Fibers Going Through Fan Pump as a Function of Pulp Consistency

Run No.	*Fiber concentration in beater, %*	*Long fibers after beating, %*	*Long fibers after going through fan pump, %*	*Loss in long fibers, %*
1705	1.5	41.5	28	32.5
1706	1.0	46	35	24.0
1708	0.7	49	36	26.5

with the water. The concentration of the stock goi onto the wire was 0.3%.

It was found that passage of the glass-fiber pap through rolls of which one or both surfaces were met gave a product containing pinholes. For this reas the couch roll was not used, and the paper could not calendered.

A woolen jacket which acted as a cushion was plac on the first-press top roll. Thus, the paper pass through rolls with felt on one side and the jacket the other. With this setup the pressure on the pap could be increased without injuring the fibers. It h been found possible to substitute a rubber roll or grani roll for the jacket-covered roll.

Since glass-fiber paper has more strength when w than when dry, handling over the drying rolls and on the reel is facilitated by keeping an appreciable moistu content in the paper. For this reason drying was reg lated so that the paper leaving the drying cylinde had a moisture content of about 50%. Subsequentl the rolls of glass-fiber paper lost moisture very rapid at room temperature, probably because of the hi degree of porosity of the paper, so that an equilibriu moisture content of about 0.1% was soon reached.

In the process of manufacture on the semicommerc mill, the details of some of the operations, such as t setting of the beater roll and the time of beating, had be varied slightly to take account of differences in t diameter of nominally identical lots of glass fibe For example, the AAA fiber in one bag averaged 0.6 mu in diameter and in another bag 0.5 mu. Such difference could materially affect the properties of t finished paper in small experimental runs, using p haps only two bags (20 lb.) of fiber. In a commerc run using 100 or more bags, for example, such va tions would tend to average out.

A Bauer-McNett pulp classifier was used by t Naval Research Laboratory to study the effect fiber length on the strength of glass fiber paper a guide to the beating procedure. This device has f compartments, each containing an agitator and screen. The screens are 65, 100, 150, and 200-mesh compartments 1 to 4, respectively. In carrying ou test, a 10-gram sample of stock is placed in compa ment no. 1 in which water is running continuou As the suspension flows through the classifier from

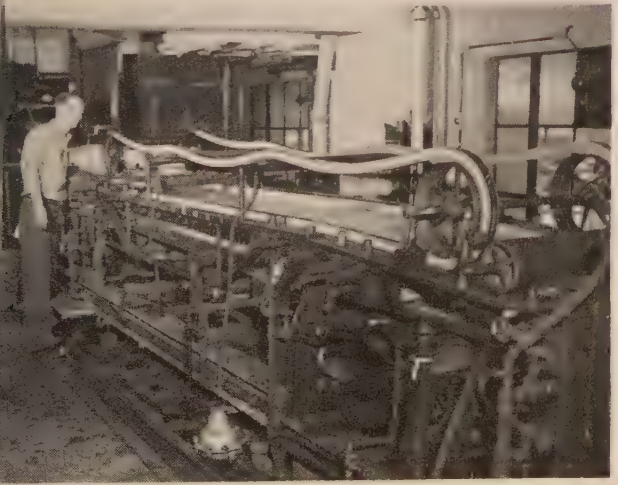
Fig. 2. Furnishing glass fibers to beater

partment to the next, the longest group of fibers is ined on the 65-mesh screen, the next longest on the mesh screen, and so on. The fibers that pass ugh the 200-mesh screen are called "fines."

sample of stock of AAA fiber as prepared for the er machine was taken from the beater. Handsheets e made from a part of the sample, and another por- of it was run through the pulp classifier. Hand- ts made from the fibers retained by the 65-mesh en were found to have a tensile strength one-fourth of the handsheets made from the original stock. dsheets made from the fibers retained on the 100, and 200-mesh screens mixed together were so k that the strength could not be measured. Hand- ts made from the fibers from the 65-mesh screen ed with those from the 100, 150, 200-mesh screens e ten times as strong as the paper from the original k.

is of interest to note that neither the paper made n the long fibers (65-mesh screen) nor that from the bined short fibers was as strong as the paper from original stock. The explanation of this is that when longest fibers were used, no short fibers were present ind the long fibers together, and when the shorter rs were used, no long fibers were present to give the er strength. With the combination of the long and rt fibers, the paper was ten times as strong as the er from the original stock because all the "fines" e removed. This proves to be an important factor the manufacture of glass-fiber paper—the more es" there are in the paper, the weaker the paper omes.

t is evident from these observations that the treat- nt necessary for glass fiber in the beater must be erent from the treatment usual for vegetable fibers. e purpose of beating vegetable fibers is in part to ise and crush them and thus increase their fibrillar ure and in part to hydrate them and gelatinize their faces. The fibrils of the vegetable fibers tend to t together and the gelatinous surfaces to adhere in paper and increase its strength. Bruising and shing the glass fibers merely tend to break them and duce shorter fibers and fines. Although it is not the pose of this report to discuss the mechanism of fiber nding in glass-fiber paper, it may be pointed out in



. 3. View of wet end of paper machine, showing the wire on which the paper is formed

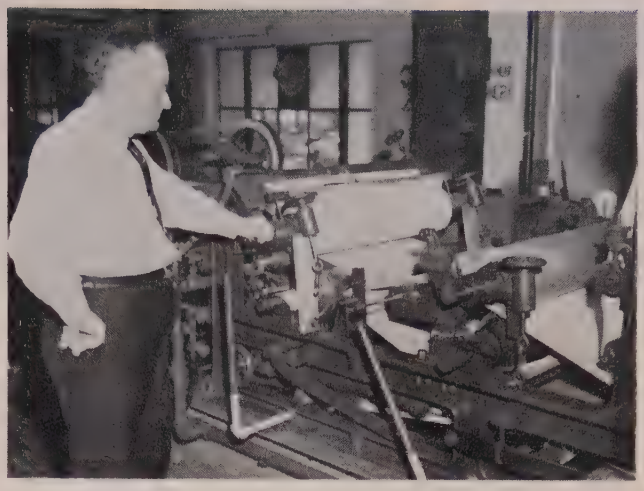


Fig. 4. First press of paper machine with jacket on top roll

passing that the all-glass fiber paper apparently owes its strength to friction between fibers rather than to the sort of entanglement and adhesion that occur in vegetable fiber papers. The coefficient of friction of the surfaces of the glass fibers, the number of contacts between fibers, and the pressure between them resulting from their elastic deformation in the structure, are important factors.

The experiments indicate that the beating of glass fibers should be conducted so as to produce a uniform suspension with as little breakage as possible.

DISCUSSION OF EXPERIMENTAL PAPERS AND TEST DATA

The conditions of manufacture of the experimental papers and their physical characteristics are given in Table II. The physical properties of the papers were tested according to the methods of the Technical Association of the Pulp and Paper Industry, with the exception of the air-resistance test. This was made with an instrument devised by the Naval Research Laboratory by means of which the pressure drop, in millimeters of water, across 100 sq. cm. of surface of the paper, with a flow of 85 liters of air per minute through it, was measured. A glass-fiber paper having an air resistance of 100 mm. has a Gurley resistance of 1.3 sec. when tested according to TAPPI Standard T 460 m.

One of the principal purposes of the experiments was to produce all-glass fiber paper with air resistance between 100 to 120 mm. The weight of the paper was varied from 40 to 51 lb. (25 × 40—500) and the thickness from 10 to 14 mils. The settings of the paper machine were obtained as follows. When the beating of the stock was completed, a 3-gram sample was taken from the beater, to determine the freeness of the stock, and mixed with water at 80°F. to a volume of 1200 cc. The freeness was determined by the number of seconds required for 1000 ml. of water to drain from the mixture. The Williams freeness tester was used. The freeness of the stock and the air resistance tests of the paper correlate; if the freeness test is high, the air resistance is high also, and vice versa. From the results of this test, the approximate amount of stock could be removed from the beater to produce a handsheet of the

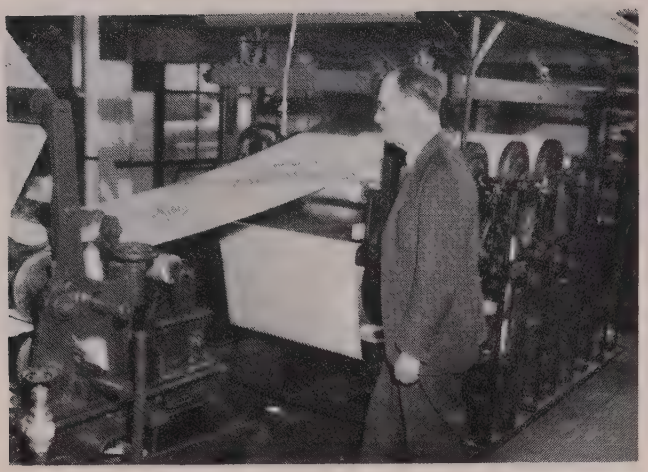


Fig. 5. View of glass-fiber paper running from second press of paper machine to driers

desired weight and thickness. The handsheet was pressed with the approximate pressure in the hydraulic press that the sheet would receive on the paper machine. The handsheet was slowly dried on the sheet drier and then tested for weight, thickness, and air resistance.

DETAILS OF MACHINE RUNS

Machine run 1660 was made with the AAA fiber (0.5 to 0.75 mu) and a concentration of the stock in the beater of 2%. At this concentration the stock was so bulky it would not circulate. It had to be propelled by hand for 20 min. before the fibers were separated. The paper was well closed and had strength enough to withstand the pull of the reel of the paper machine.

Run 1661 was made from AA fiber (0.75 to 1.5 mu). The concentration of the stock was reduced to 1.5% and it circulated in the beater without aid. This paper was not so strong as the AAA fiber paper, run 1660, but it was strong enough to withstand the pull of the reel. The approximately 20-mil sheet had an air resistance of 80 mm. or 4 mm. per mil. To get the same air resistance from the AA fiber as was obtained from the AAA, the paper would have to be $2^1/_2$ times as thick.

Run 1662 was made from the A fiber (1.5 to 2.5 mu). These fibers are coarse and hard to handle. After running the A fiber over the machine, the bottom of the sand trap was covered with small particles of glass. The finished paper was weaker than the AA fiber paper, run 1661. It could not withstand the pull of the reel and had to be reeled by hand.

Run 1663 was made from the B fiber (2.5 to 3.75 mu). These fibers have characteristics similar to the A fiber. The paper was so weak that when thoroughly dried, it disintegrated on handling; however, when there was 40 to 50% water in the paper, it was reeled with the aid of a conveyor, without any trouble.

It can be concluded from the foregoing experiments, that as the diameters of the glass fibers decrease, the paper increases in strength, air resistance, and presumably in value as an air filter.

The following additional runs were made from AAA fiber: 1705, 1706, 1707, 1708, 1709, 1710, and 1712.

The effect of beating at different concentrations was obtained by comparing run 1705 (1.5% concentration) with run 1660 (2% concentration). The long fibers were increased 50%, and the "fines" reduced 13% by

Table II. Comparative Data on Papers Made from Glass Fibers

Run No.	Beating time, min.	Beater furnish, grade of fiber	Fiber concentration in beater, %	Basis weight (25 × 40—500), lb.	Thickness, in.	Tensile strength Machine direction, lb./in.²	Tensile strength Cross direction, lb./in.²	Tensile strength Machine direction, lb./in. width	Tensile strength Cross direction, lb./in. width	Tearing resistance Machine direction, g.	Tearing resistance Cross direction, g.	Air resistance[a], mm.	Air resistance[a], mm./mil.	Density, g./cm.³	Smoke filtration efficiency[b]
1660	20	100% glass fiber AAA	2.0	42.0	0.0105	34	23	0.36	0.24	9	7	103	10	0.230	Good
1661	15	100% glass fiber AA	1.5	91.5	0.0190	Paper too weak to test						82	4.3	0.268	No test
1662	15	100% glass fiber A	1.5	173.0	0.0330	Paper too weak to test						45	1.4	0.292	No test
1663	15	100% glass fiber B	1.5	220.0	0.0450	Paper too weak to test						22	0.5	0.272	No test
1705	15	100% glass fiber AAA	1.5	48.4	0.0120	42	37	0.50	0.44	32	33	120	10.0	0.224	Good
1706	25	100% glass fiber AAA	1.0	45.9	0.0116	54	36	0.63	0.42	25	29	100	8.6	0.220	Good
1707[d]	25	100% glass fiber AAA	1.0	48.7	0.0129	42	43	0.54	0.56	32	27	116	9.0	0.209	Poor
1708	35	100% glass fiber AAA	0.7	50.6	0.0137	34	27	0.47	0.37	24	25	123	9.0	0.205	Fair
1709[d]	25	100% glass fiber AAA	1.0	51.4	0.0120	47	35	0.60	0.47	27	27	90	7.0	0.228	Poor
1710	90	100% glass fiber AAA	1.0	46.4	0.0147	43	34	0.63	0.50	36	38	103	7.0	0.175	Fair
1712	90	100% glass fiber AAA	1.0	43.8	0.0129	43	36	0.56	0.46	31	32	129	10.0	0.174	Good

[a] Pressure drop across 100 sq. cm./min., measured in mm. of water.
[b] As graded by tests at Naval Research Laboratory.
[c] Diameter of fibers, mu: AAA 0.5 to 0.75, AA-0.75 to 1.5, A-1.5 to 2.5, B-2.5 to 3.75.
[d] Many pinholes present. Beater roll was on bed plate instead of 0.008 in. off as in other runs.

ucing the concentration from 2% down to 1.5%, l the tensile strength was increased about 42%.

The results obtained from run 1705 were so promising t the concentration of the beater stock for machine 1706 was further reduced to 1%. Figure 1 shows t there was over 100% increase in long fibers over 2% concentration and a reduction of $18^1/_2$% in s. The tensile strength was increased by 65%.

Run 1707 was a duplicate of 1706 with the exception he beater roll setting. Up to this run the roll setgs were 0.008 in. off the bedplate. For run 1707 roll was lowered to the plate. The resultant sheet s well closed. Table II shows that the physical propies of this paper were as good as the previous runs. wever, the filtering efficiency of paper from this run s poor. Examination of the paper showed this to due to numerous pinholes.

Run 1708 was made with the concentration in the ter reduced to 0.7% to determine if this further uction would result in more long fibers. Figure 1 ws that the percentage of long fibers was compale to that of the 1% concentration and that nothing s gained by the further reduction.

Run 1709, a duplicate of 1707, was made in order to ertain if the beater roll settings were the cause of pinholes in 1707. Table II shows the filtering iciency of paper from run 1709 was poor, and this in was caused by pinholes. This led to the conclun that glass fibers could not be crushed anywhere in manufacture of paper without lowering the filtering iciency of the paper.

In machine runs 1710 and 1712 the beater roll was ered to 0.04 in. from the bed plate. With this roll ting it took 90 min. to separate the fibers. Comring the test data with previous runs, there was not ugh improvement in the physical properties to rrant the long beating time.

ADVANTAGES OF THE PAPER

The results of tests of the papers as air filters, made the Naval Research Laboratory and reported qualively in the last column of Table II, showed the -glass paper to be particularly valuable for gas masks l respirators used by fire fighters, industrial and dical workers, and military personnel. In a test h smoke-filled air only 1 particle of smoke in 100,000 netrated the paper in a gas mask, and this high effi-

g. 6. Glass-fiber paper running from driers of paper machine over calenders onto reel

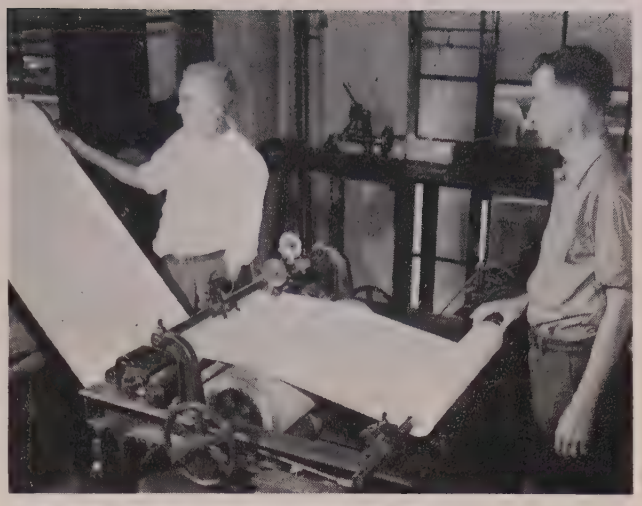


Fig. 7. Rewinding the glass-fiber paper

ciency was attained with no increase in the normal breathing resistance. No doubt the paper would be equally valuable in some other types of air filters, and it should be suitable for the filtration of liquids.

Heretofore the most efficient commercial air-filter paper was made from asbestos fibers (*2*) mixed with cellulose fibers. The Bolivian "blue" asbestos is better for the purpose than the asbestos mined in America or Canada. Asbestos has many disadvantages: Only 300 or 400 lb. of usable fibers can be obtained from a ton of asbestos as mined; the breaking down, dusting, and washing of these fibers require much time; and the distance the asbestos has to be shipped adds to its expense. The glass fibers are manufactured in the United States, and the fibers are clean and ready for the beater. The new product is more effective than the asbestos type for air filtration. Also the glass-fiber paper will not support mildew growth as will the asbestos-cellulose paper under adverse conditions. The glass fibers can be made smaller in diameter than those of the asbestos. The smaller the fiber diameter, the more efficient is the filter paper.

The high resistance of the paper to heat, moisture, mildew and other microorganisms is a decided advantage over some other types of filtering media for some uses.

The paper should have extensive use in electronics (*3*) and other electrical equipment because of its excellent insulating characteristics and high resistance to heat. The present top operating temperature of capacitors is about 85°C., because the kraft paper used in them will not withstand higher temperatures. The glass-fiber paper can withstand a temperature of 200°C. and even higher. With its use, smaller capacitors can be manufactured.

Another possible use of the paper is as a reinforcing material in plastic laminates.

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Methods of Checking Wood and Chip Quality

J. M. RALSTON and MAURICE KLEIN

Wood quality counts are made at Union Bag & Paper Corp. to determine trends in the quality of new receipts and to attempt to explain variations in operations. Each bolt in a sample is examined and classified according to definitions set up for each of the various types of undesirable wood. A description of the procedure for making wood quality counts is included. Chip size distributions are made with the Williams Standard chip screen on samples taken each shift at the chip bins. These size distributions are useful in aiding the operating department to control wood room operations. Data from tests on effect of knife angle and knife settings on chip size are included.

THE accurate determination of wood quality and chip quality in a modern paper mill should serve as an aid in explaining variations in pulp yields and paper quality and in pointing out the source of operational difficulties in the wood room and pulp mill. It is the purpose of this paper to discuss the methods used for checking wood quality and chip quality at Union Bag & Paper Corp.

Before discussing the method for checking wood quality used at Union Bag, it might be interesting to illustrate how small variations in wood quality, chip quality, or pulping operations may affect wood costs. In a mill producing 1000 tons of pulp a day an increase in wood requirements of 0.01 cord per ton of pulp would increase the annual cost about $60,000 at the present cost of wood. Thus, the use of methods for controlling these variables could materially benefit a company. Although wood consumption at Union Bag has been decreased, it is difficult to correlate this decrease with wood and chip quality measurements because of the numerous other variables. However, better control of wood deterioration on the yard, more emphasis on wood quality and chip quality, and better pulp mill operations have probably all contributed to this decrease. As the demand for wood increases in the south, wood quality may deteriorate. If this proves to be the case, improved methods for determining wood quality would be desirable to help to determine the cause for variations in wood consumption.

METHODS OF CHECKING WOOD QUALITY

Wood quality measurements at Union Bag are made to determine trends in the quality of new wood receipts and to aid in determining if operational difficulties in the pulp and paper mills are caused by poor wood quality.

Wood quality counts are made by the mill technical department and are independent of the scaling of new wood. In fact, the classification of undesirable wood for the quality counts differs considerably from the procedure used by the scalers for culling subquality wood. In addition to differences in definitions of undesirable types, the wood culled by the scale includes only those cull types above a certain acceptab minimum per car. The wood quality counts are r ported on the basis of the number of bolts count whereas wood culled by the scalers is reported on t volume basis.

An extremely important point in checking wo quality is a clear definition of each type of undesirab wood. Definitions of undesirable types may va from mill to mill according to existing condition Our definitions of undesirable wood types and so comments on the effect that each has on yield, pu quality, and operations are given below.

Old Wood. Wood which shows visible signs of decay such l loosening or loss of bark and discoloration or decay at the b ends is classified as old wood. No attempt is made to classf the extent to which old wood has decayed. The use of old wo decreases pulp yields and strength, increases chemical consum tion, and may increase the production of undersized chi Figure 1 shows an old bolt and a freshly cut bolt for comparison

Crooked and Limby Wood. Wood having large enough lim projecting from the trunk or sufficient curvature to cause a vo space of 3 in. or more when stacked in ricks for shipment is clas fied as crooked and limby wood. In addition to reducing t solid wood volume of shipments, crooked and limby wood believed to cause difficulty in chipper operations and to yi poor quality chips from which a lower yield of acceptable pu is obtained. Wood having limbs which have been trimm sufficiently to prevent voids is not included in this categ Figure 2 is a photograph of a crooked bolt with a protrud limb. A straight bolt is included for comparison.

Small Wood. Wood having a diameter of less than 4 in. ins the bark at the small end is classified as small wood. Sm wood frequently causes choking of the barking drums and b conveyors. Solid wood volume per cord of small wood is l than that of wood of normal diameter. In addition it is felt t chips produced from small wood are inferior in quality. Figu shows two small bolts with a normal size bolt for comparis

Catface Wood. Any wood that has sections of bark cut t collect the tree gums is classified as catface wood. This typ undesirable wood is further subdivided into burnt catfaces catfaces containing metal. The use of catface wood, particula that with metal, increases the cost of the chipping operation may yield poorer chips. Burnt catfaces also contribute to a di sheet, if not properly cleaned. However, the use of catfa may contribute to a higher yield of sulphate soap and turpenti Figure 4 shows two catface bolts, one with considerable bu areas.

It is the purpose of our quality counts to determ the amount of each undesirable type (as previou defined) that is received in our new wood receip Approximately 1% of our new receipts or about 43,0 bolts each month are classified by observation of t individual bolts as they are conveyed to the bark drums.

Quality counts are made about 4 or 5 days each we and require about 2 hr. each day. It has been fou that the quality of new receipts from different locati varies considerably. Wood is received by truc barges, flat cars, and box cars. As these four ty of carriers often handle wood from different locatio

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Fig. 1. Old wood

number of bolts counted from each type of carrier ivided in proportion to the percentage of receipts n each type of carrier. No attempt is made to sify yard wood since only flat car receipts are sent he yard and no discrimination is made in the dis- tion of these receipts to the process or yard. Ad- edly larger counts would be more accurate; never- ess, it is felt that the data obtained represent y accurately the trend in quality of our new re- ts from month to month.

o facilitate the actual counting operation, a nine- multiple counter is used. A picture showing the nner in which wood is counted with this counter iven in Fig. 5. This enables the wood quality ker to classify the logs rapidly as they pass on the veyor without having to tabulate by hand the ous undesirable types. No other mechanical is used for the actual classifications. Small wood been classified with a 4-in. fork several times. wever, because this practice is slow and somewhat gerous, it is not used regularly. With a little tice, the wood quality checker can classify small d accurately by eye. Our wood quality checkers e been instructed to classify only those bolts which be seen sufficiently to determine their classifications.

Fig. 2. Crooked and limby wood

Fig. 3. Small wood

Thus, bolts hidden beneath other logs are not included in the counts. Because the classification of undesirable wood is a matter of judgment, the training of new wood quality checkers requires considerable time. Nearly all of our wood counts have been made by one individual since they were started in 1943. Thus, it is felt that our counts are probably more comparable than if they had been made by several individuals.

Our quality counts, which are reported on the basis of the number of bolts counted, indicate that approximately 20% of our new receipts are undesirable. It is estimated that the undesirable wood receipts would amount to about 11% of our new receipts if reported on the volume basis. Also on the volume basis, the quality counts indicate that about 2.5% of the new receipts or 21% of the undesirable wood are small wood.

Our quality counts have definitely reflected the efforts of our wood procurement department to have metal and burnt areas removed from catfaces and to have protruding limbs cleaned from the limby bolts before shipment. To illustrate, prior to December, 1948, about 3% of our new receipts were classified as crooked and limby wood. Since that time, our receipts of crooked and limby wood have been reduced to about

Fig. 4. Catface wood

Fig. 5. Wood classification

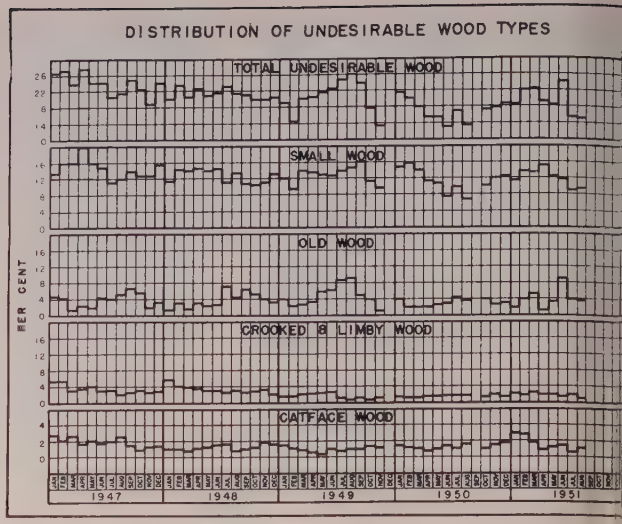

Fig. 6

1.5%. Over the same period of time our receipts of uncleaned catfaces have been cut in half. This indicates the cooperation existing between the woodlands division and the wood dealers.

Our quality data are presented to management in a monthly progress report giving a tabulation of the quality of new truck and rail receipts as compared to the previous 6 mo. and also in a combined graphical presentation showing each undesirable type for the previous 4 years. Figure 6 shows graphically the undesirable receipts for 1951 and the past 4 years. The quality data are also included in a monthly wood operations report and sent to our woodlands and New York offices. At times we have wondered how frequently our quality data are referred to. When it was impossible to obtain counts for 1 mo. because of wood yard operations and personnel shortage, there were numerous requests received for the data and a renewed emphasis from management was placed on the importance of the data.

METHODS OF CHECKING CHIP QUALITY

The chip quality at Union Bag is determined daily with a Williams Standard chip screen having square mesh screens of $^1/_8$, $^1/_4$, $^1/_2$, $^3/_4$, and 1-in. openings. This method is used in place of the TAPPI Standard T 16 m-47. One 10-lb. sample is taken on ea shift from the chip conveyor belt at the chip bins such a way as to take a cross section of the flow chips. Because of the variability of chip quality, so other means of obtaining a more representative samp such as by quartering a larger sample, would be d sirable. However, as chips to our bins have be screened to remove oversize chips and sawdust, lar variations in the quality of chips from the chippe are reduced. It is felt that the monthly average our chip size distribution indicates trends in ch quality. Our daily chip quality data are also us to help control wood room operations. As yet, correlation of these data with pulp yields has be made because of the numerous variables involved. may be possible to determine the optimum chip s in a new pulping laboratory recently installed.

The monthly chip size distribution is report graphically to management in the monthly wood ope tion report. Figure 7 shows the chip size distributi for this year to date and the past four years. "Go Chips" are the summation of all fractions excludi the fraction 1 in. and over and the sawdust fracti (passing $^1/_8$ in.).

Considerable work has been done to try to det

Table I. No. 4 Chipper Performance Tests

Period	*1*	*2*	*3*
Chipper disk—no. of knives	8	10	10
Date of test	4/1-9/29/48 1/28-3/22/49	1/4-1/27/49 3/23-7/26/49	3/23-7/26/49
Knife setting, in.	$^{15}/_{32}$	$^{11}/_{32}$	$^7/_{16}$
No. of 8-hr. performance tests	28	16	9
No. of classifications	413	214	113
Av. age of wear plates, mo.	2.7	1.6	1.8
Chips retained on 1-in. screen, %	14.2	19.6	15.8
Chips retained on $^3/_4$-in. screen, %	30.7	36.7	36.6
Chips retained on $^1/_2$-in. screen, %	30.2	26.8	30.0
Chips retained on $^1/_4$-in. screen, %	21.0	14.4	15.5
Chips retained on $^1/_8$-in. screen, %	3.2	1.9	1.7
Sawdust, %	0.7	0.6	0.4
Good chips, %	85.1	79.8	83.8

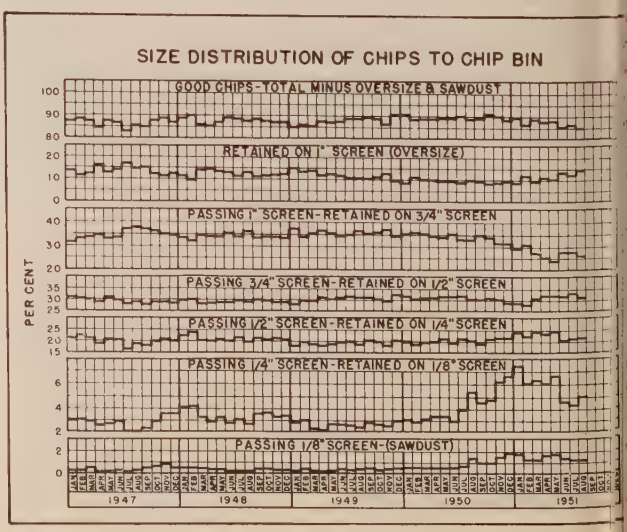
SIZE DISTRIBUTION OF CHIPS TO CHIP BIN
GOOD CHIPS - TOTAL MINUS OVERSIZE & SAWDUST
RETAINED ON 1" SCREEN (OVERSIZE)
PASSING 1" SCREEN - RETAINED ON 3/4" SCREEN
PASSING 3/4" SCREEN - RETAINED ON 1/2" SCREEN
PASSING 1/2" SCREEN - RETAINED ON 1/4" SCREEN
PASSING 1/4" SCREEN - RETAINED ON 1/8" SCREEN
PASSING 1/8" SCREEN - (SAWDUST)
PER CENT
1947
1948
1949
1950
1951

Fig. 7

r the effect of chipper conditions on chip quality. e of the variables affecting chipper performance the age of the chipper wearplates, knife settings, e sharpness, chipper loading, and wood quality. rial chipper performance tests are usually made running size distributions on samples from the chip- taken at half-hour intervals over a period of 8 hr. is period was chosen to cover the complete range of e sharpness, as knives are usually changed at the nning of each shift and may be sharpened by hand ntermediate times. No attempt is made to control d quality during chipper performance tests al- ugh this is probably an important variable. It indicated by some of our tests that increased chip- loading tended to increase the production of over- chips. Therefore, to determine the effect that ations in other conditions may have on size dis- ution, samples for such tests are taken during ods of average loading as determined by obser- on of the load. Because of normal variations in size distribution due to the numerous uncontrolled ables, considerable data must be collected to draw ible conclusions.

ome of the results of our special chipper performance s are given below. It was found that knife sharp- was maintained longer by increasing the knife ing angle from 38 to 40° without any deterioration hip quality. As a result, our knife cutting angles maintained at 40°. The effect of decreasing chip- knife settings was investigated on our no. 4 chipper g eight and ten-knife disks. Data on the chip size distribution from this chipper with the eight and ten-knife disks are given in Table I. It will be noted that the size distribution of chips from the ten-knife disk was considerably poorer than from the eight-knife disk with the same knife setting. After the knife settings of the ten-knife disk were reduced from $^{15}/_{32}$ to $^{7}/_{16}$ in., the size distribution of the chips from the chipper was approximately the same as for the eight-knife disk with a $^{15}/_{32}$-in. knife setting. The data also indicate that chip size distribution deteriorates with the age of the chipper wearplates.

Density, moisture, and hollocellulose are also determined on our routine samples by slight modifications of the TAPPI procedures. Insufficient data have been obtained at the present time to evaluate their usefulness.

Although the accurate measurement of wood quality and chip quality is difficult, it is believed that these methods have been useful in controlling operations. The formulation of adequate definitions of undesirable types of wood, according to the conditions existing at a mill, is extremely important in the checking of wood quality. The actual procedure for determining the wood quality depends upon the manner in which wood is handled at a mill. The recent installation of a new pulping laboratory at Union Bag should enable us to determine more accurately the effect that various undesirable wood types and variations in chip quality have on pulp yields, paper quality, and mill operations.

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The Properties of High Yield Kraft Pulps Produced at Bathurst

J. McK. LIMERICK

h increased yield and conservation of the natural re- rces constantly in mind the Bathurst Power and Paper are currently operating their kraft mill at a pulp yield 62%. A contributing factor has been brown stock ning according to the Sutherland High Yield Process, ereby kraft pulp in the presence of hot black liquor is ned before passing to the brown stock washers. Pulp resenting current operating practice having a chlorine mber of 16.2 and a yield of 62% and a pulp from a trial of 19.2 chlorine number and 64% yield have been eval- ed for physical and chemical characteristics. These e been compared with a commercial grade not produced hot refining having a chlorine number of 2.9 and yield 47%. The strengths of the high yield pulps compared y favorably. The two Bathurst grades were bleached in laboratory for experimental purposes, by means of a -stage process and compared with a commercially ached grade. Again the results compared well and the % yield from the wood for the bleached high yield pulp uld have to be evalated against high bleach consump- n. High yield pulps produced by this process have high tial strength due to the brown stock refining. As they become rougher they require more refining to arrive at any given freeness, but the strengths at any freeness compare very favorably and high yield pulps of the type described are producing a kraft linerboard of good quality.

With wood costs representing such a large portion of the value of pulp and paper and with the price of wood rising, it becomes increasingly important to strive for improved yields from the raw material. A glance at Table I will make this readily apparent. It is based on wood having a density of 0.35 and priced for convenience at $25 per cunit. The table gives the savings to be accomplished per ton of pulp by increasing the yield to values over 50%.

Table I. Savings in Wood Costs by Increased Yields Wood at $25 per Cunit—Density 0.35

Per cent yield, O.D. wood—O.D. pulp	*Savings per ton of pulp, dollars*
50	0.00
55	4.10
60	7.70
65	10.50
70	13.10

cK. Limerick, Research Director, Bathurst Power and Paper Co. Ltd., urst, N. B.

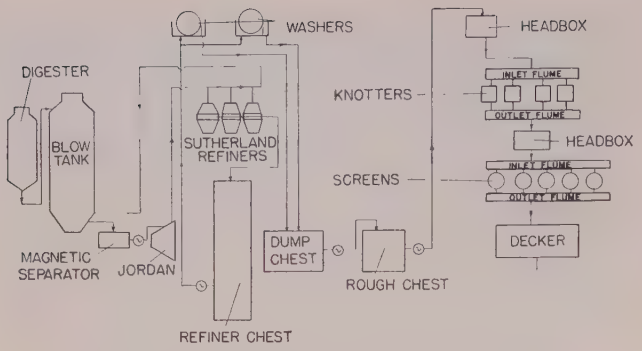

Fig. 1. Flow diagram of kraft pulp mill

Not only does an increased yield result in a saving in wood cost but it increases the production from a given number of digesters and also gives rise to a conservation of the natural resources of the country. For several years the staff of the Bathurst Power and Paper Co. have concentrated a good portion of their development efforts on higher yields in the kraft process. In 1945 brown stock refining known as the Sutherland High Yield Process was placed in commercial operation and since that time yields have gradually been increased. This is a report on some physical and chemical properties of a pulp having a yield of 62%, which is now current practice, and also similar results on an experimental run of pulp of 64% yield. Both types were bleached in the laboratory and evaluations are also given on the bleached pulps.

GENERAL METHOD OF PULP PRODUCTION

The layout of the kraft pulp mill and brown stock refining system was described in detail by Allo (*1*) in 1947. It has not changed substantially since that time and is shown in Fig. 1.

Briefly, the pulp from the digesters is blown into a large blow tank. From this tank the stock passes through a magnetic separator to remove tramp metal, through a pump, and then to a jordan which acts as a chip breaker and also as a further protection to the refiners in catching any tramp metal which was not removed at the magnetic separator. The pulp then passes to three Sutherland brown stock refiners. The refiners, which are operated in parallel, are the heart of the Sutherland High Yield Process. They refine the pulp in the presence of hot black liquor and defiber the coarse fractions, thus making it possible to utilize pulps cooked to a lesser degree and having a high yield.

From the brown stock refiners the pulp drops to the refiners chest, from which it is pumped to two Nyman-

Table II

	Cl No. 2.9	*Cl No. 16.2*	*Cl No. 19.2*
Yields Calculated on Dry Wood			
Unscreened yield, %	...	62	64
Lignin in unscreened pulp, %	...	9.3	11.4
Flat screen rejects, %	...	1.7	3.1
Screened yield, %	47	60.3	60.9
Lignin in screened pulp, %	1.27	8.0	10.1
Bleached screened pulp yield, %	42.8	51.1	50.0
Yields Calculated on Unbleached Pulp			
Lignin in unscreened pulp, %	...	15.0	17.8
Flat screen rejects, %	...	2.8	4.8
Screened yield, %	...	97.2	95.2
Lignin in screened pulp, %	2.7	13.2	16.6
Bleached screened yield, %	91	84.7	82.0

Table III. Chemical Analyses of Screened Unbleached Pulps

	Cl No. 2.9	*Cl No. 16.2*	*Cl No. 19.*
Lignin, %	2.7	13.2	16.6
Holocellulose, %	96.7	85.4	83.0
Alpha-cellulose, %	87.0	72.7	70.7
Hemicellulose, %	9.7	12.7	12.3
Pentosan, %	8.0	9.6	9.1

Swenson brown stock washers operating in parall From this point the stock passes through knotte screens, and thickeners and hence to the paper m chines. It should be mentioned that all the pu coming from the digesters is accepted stock under t operating conditions used at Bathurst. Linerboa receives only screened pulp and any rejects are refin and go to the system supplying the corrugating boa machine.

PRODUCTION OF PULPS DESCRIBED IN THES EXPERIMENTS

In the previous description of the Sutherland Hig Yield Process at Bathurst using brown stock refinin the pulp yield was 58% and the Roe chlorine numb was 13 in regular operation. A few months later trial run was made of pulp having a chlorine number 16.2. The pulp was shipped to the Pulp and Pap Research Institute of Canada and evaluated by Willia Lodge and his associates. These are the results whi are reported later in this paper under the heading pulp having a chlorine number of 16.2.

At the time of the trial run it was not possible to ru pulp of 16.2 chlorine number continuously in co mercial operation. However, changes which we indicated at the time have since been made. Refin disks have been changed from 42 to 48 in. and the di design has been greatly improved. A new screeni system has been placed in operation and cooking co ditions have been modified considerably. The res is that pulp having a chlorine number of 16 to and a yield of 62% is now standard commercial practi at Bathurst. The active alkali charge per air-d ton has been decreased from 460 lb. as reported for 58 yield to 430 lb. for 62% yield.

Once yields of 62% had become established practic studies were immediately under way to examine t possibilities of increasing the chlorine number ev further. In August, 1950, a trial run was made of o digester of pulp. The active alkali charge was furth lowered to 420 lb. per ton and the cooking time r duced by 20 min. When blown the pulp had a chlori number of 19.2.

On this run the three Sutherland refiners were s up very hard and operated at a reduced capaci The pulp ran in a normal way over the brown sto washers and passed through the screening system in t same manner as a pulp of regular cooking degree. should be made clear, however, that stock of this coo ing degree is not being run in regular operation present. In order to refine the pulp so that it could

Table IV. Chemical Analyses of Bleached Pulps

	Cl No. 2.9	*Cl No. 16.2*	*Cl No. 1*
Alpha-cellulose, %	87.1	85.7	86.4
Hemicellulose, %	12.9	14.3	13.6
Pentosan, %	8.0	10.5	8.8

Table V. Screen Analysis

	Unbleached		Bleached	
	Cl No. 16.2	Cl No. 19.2	Cl No. 16.2	Cl No. 19.2
ness (Canadian), ml.	718	717	675	679
ned 28-mesh	73.5	82.6	73.9	78.7
ned 48-mesh	11.4	6.6	12.6	8.3
ned 100-mesh	4.8	2.3	5.6	4.1
ned 200-mesh	2.9	1.5	3.0	1.1
d 200-mesh	7.4	7.0	4.9	7.8

lled by the screens, the refiner disks had to be well up and operated at reduced capacity so that it ld not have been possible to keep the paper ma- es operating at the reduced rate of pulp production. rever, with the increased hot refining the pulp d well throughout the system and produced a good le of linerboard.

supply of pulp from this run was saved and evalu- for physical and chemical properties in the Re- ch Division of the Bathurst Power and Paper Co. s referred to in the following part of the paper as having a chlorine number of 19.2.

he wood species used during the specific runs de- bed in this report may be of interest. The pulp ing a chlorine number of 16.2 was made from wood sisting of 70% white spruce and 30% balsam and ing a density of 0.360. The stock of 19.2 chlorine mber was pulped from 82% balsam, 16% white ce, and 2% black spruce. The wood density 0.338.

ortions of both runs having chlorine numbers of 2 and 19.2 were bleached on a laboratory scale. e unbleached pulp was chlorinated at 3% consistency 15°C. for 45 min. with 90% of the chlorine re- rement. It was then given an alkali extraction with .5% solution of caustic soda at 4% consistency. A her chlorination with 10% of the chlorine demand s carried out under the same conditions as before. other alkali extraction followed. At this stage a sh with sulphur dioxide was given and the pulp htened considerably due, it is thought, to the re- val of iron contamination. The final bleach was ried out in two stages with an intermediate wash ng 0.5% chlorine (based on pulp) as calcium hypo- orite in each stage. The consistency was 5%, the nperature 35°C. and the pH 10.5. The chlorine s not completely exhausted in each stage. The al treatment was a wash with sulphur dioxide and the lp brightness was 82 G.E. in each case.

In short, what the above procedure means is that the lps of 16.2 and 19.2 chlorine number, respectively, re bleached to 82 G.E. brightness with about 17 and % of chlorine. The chlorine consumption was gh and multiple stages were required for bleaching. cause commercial bleaching is not carried out at thurst the bleached pulps were evaluated more as a tter of academic interest and no bleaching costs

ble VI. Beater Test—Unbleached Kraft—Cl No. 2.9

ting ne, in.	Free- ness, ml.	Den- sity	Burst factor	Tear factor	Breaking length, in.	Stretch, %
0	681	0.650	32.1	250	7,100	3.4
5	630	0.710	74.0	138	12,100	3.3
0	566	0.724	87.8	126	13,700	3.3
5	479	0.750	94.8	115	14,600	3.5
0	370	0.768	93.3	108	14,500	3.6
5	266	0.781	87.2	103	14,500	3.7

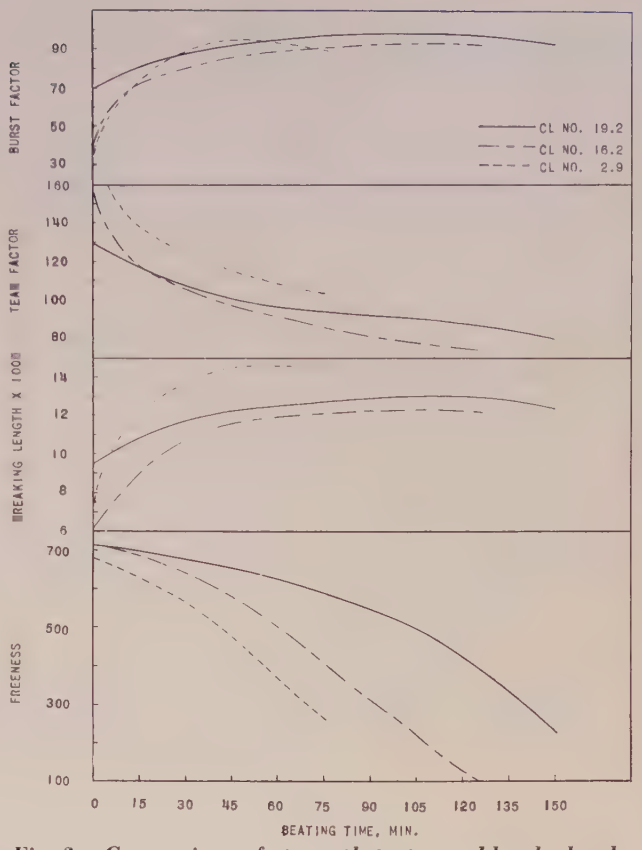

Fig. 2. Comparison of strength tests—unbleached pulp

were calculated. However, in view of some of the strengths obtained and the yield of bleached pulp obtained from the wood, some mills may have an interest in evaluating high chlorine consumption against pulp yield.

EXPERIMENTAL PROCEDURE

The pulp from each of the runs was sampled at the discharge side of the brown stock washers and some of the analyses were conducted on it. The beater test, chemical analysis, and bleaching were carried out on the pulp after it had been screened on a laboratory flat screen having a 0.007-in. cut plate. A check beater test on unscreened stock gave the same results.

All beater tests were carried out on slush stock which had never been dried. The Valley beater was used exactly as outlined in TAPPI Standard T 200. Test sheets were made according to TAPPI Standard T 205 and tested according to TAPPI Standard T 220.

All other determinations were carried out according to TAPPI Standards.

In order to have a comparison in certain respects with a commercial bleached and unbleached kraft of lower chlorine number (but not produced by brown stock refining), a supply of these grades in slush form was obtained from another mill in New Brunswick

Table VII. Beater Test—Unbleached Kraft—Cl No. 16.2

Beating time, min.	Free- ness, ml.	Den- sity	Burst factor	Tear factor	Breaking length, in.	Stretch, %
0	718	0.441	36.1	155	6,120	2.7
10	701	0.510	68.3	125	8,190	2.9
40	606	0.606	84.5	99	11,400	3.4
60	507	0.642	88.5	92	11,880	3.6
80	376	0.666	91.2	84	12,150	3.6
95	283	0.685	92.5	82	12,260	3.7
110	184	0.690	93.4	77	12,290	3.6
125	103	0.699	93.2	74	12,230	3.5

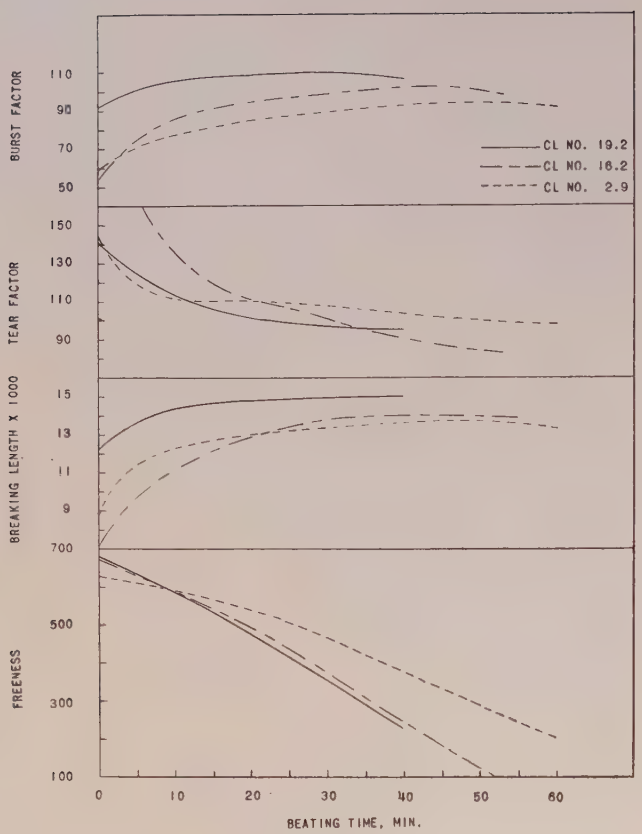

Fig. 3. Comparison of strength tests—bleached pulp

which manufactures bleached kraft pulp. These samples were evaluated under the same conditions as above in the Bathurst Research Division and the results are reported under the heading of pulp having a chlorine number of 2.9. The unbleached yield of 47% as given for this pulp was the figure supplied by the manufacturers. It is considered by some to be on the high side for pulp of 2.9 chlorine number.

The yields, lignin contents, and rejects on the laboratory 0.007-in. flat screen for the three pulps are given in Table II. The calculations are shown as percentages of dry wood and unbleached pulp.

Chemical analyses for the unbleached pulps are shown in Table III and for the bleached in Table IV. The chlorine numbers in the headings are those of the pulps after the brown stock refiners. After laboratory screening they were 14.5 and 18.4.

Fiber classifications carried out according to the method using the Johnston screen classifier for the unbleached and bleached pulps (unbeaten) from the two Bathurst runs are shown in Table V. The pulps of chlorine number 2.9 were not classified.

The results of the complete beater tests and strength values of the three unbleached pulps are shown in Tables VI, VII, and VIII and for the bleached grades in Tables IX, X, and XI. For comparative purposes

Table VIII. Beater Test—Unbleached Kraft—Cl. No. 19.2

Beating time, min.	*Freeness, ml.*	*Density*	*Burst factor*	*Tear factor*	*Breaking length, in.*	*Stretch, %*
0	717	0.575	69.6	128	9,460	3.6
30	679	0.651	88	107	11,800	3.9
60	624	0.672	94	96	12,500	4.1
90	549	0.718	98	92	12,850	4.2
120	425	0.751	98	88	13,100	4.2
150	235	0.764	93	80	12,400	4.2
180	98	0.789	90	77	12,200	4.2

Table IX. Beater Test—Bleached Kraft—Cl No. 2.9

Beating time, min.	*Freeness, ml.*	*Density*	*Burst factor*	*Tear factor*	*Breaking length, in.*	*Stretch, %*
0	630	0.700	59	144	8,700	3.5
10	591	0.747	77	111	12,300	3.4
30	470	0.764	87	108	13,300	3.6
45	333	0.769	93.1	102	13,700	3.6
60	202	0.789	91.4	98	13,300	3.4

graphs showing the essential information are given in Figs. 2 and 3.

As a further means of comparison the strength values at zero beating time and at 450 freeness have been taken from the beater tests and set forth in the form of tables. Those for the unbleached pulps are shown in Table XII and for the bleached in Table XIII.

DISCUSSION OF RESULTS

One of the first striking features is the high initial bursting strength for the high yield, high chlorine number pulps. This should not be surprising. It must be remembered that the pulps have actually been refined after the blow tank and the burst has been increased. In fact, the stock has received some pre-beating so that the so-called initial or unbeaten bursting strength is greater than normal for a pulp of high chlorine number.

At any given freeness and at the maximum the strengths compare very well with pulps of lower yield.

Table X

Beating time, min.	*Freeness, ml.*	*Density*	*Burst factor*	*Tear factor*	*Breaking length, in.*	*Stretch, %*
0	675	0.544	53.2	202	7,110	3.4
10	591	0.629	85.8	136	11,190	3.6
20	497	0.694	94.9	111	12,750	3.6
30	375	0.725	98.6	101	13,850	3.8
37	285	0.741	101.2	93	13,880	4.0
42	221	0.758	102.4	89	14,010	4.2
52	101	0.775	98.9	83	13,840	4.0

It is characteristic of these high yield pulps that a few points of freeness drop mean quite an increase in bursting strength. The Bathurst Power and Paper Co. has accumulated considerable data on this point both in the mill and particularly in the pilot plant and it is not at all uncommon to see the burst factor tripled when the freeness has been lowered from 750 to 700. These pulps very frequently reach their maximum burst between 500 and 600 freeness and from this point beating seems to make no difference. This should not be taken to mean that they are easy-beating pulps. In fact the reverse is true and it takes more refining to reach a given freeness as the pulp becomes higher in chlorine number. This will be apparent from the times to reach 450 freeness in Table XII.

Under the present freight classifications it is unfortunately true that the bursting strength test, even though it is not a good measure of box performance is the sole strength test required for linerboard. For this reason burst has been especially mentioned, but

Table XI

Beating time, min.	*Freeness, ml.*	*Density*	*Burst factor*	*Tear factor*	*Breaking length, in.*	*Stretch, %*
0	679	0.726	92	141	12,200	3.3
10	586	0.768	106	114	14,400	3.3
20	471	0.786	109	102	14,800	3.4
30	352	0.800	110	97	14,900	3.4
40	225	0.810	106	95	15,000	3.5

Table XII. Comparison of Unbleached Pulps

	At zero beating time			At 450 freeness		
	Cl No. 2.9	Cl No. 16.2	Cl No. 19.2	Cl No. 2.9	Cl No. 16.2	Cl No. 19.2
·ss (Canadian),						
	681	718	717	450	450	450
·to freeness, min.	0	0	0	49	69	116
tfactor	32.1	36.1	69.6	94.4	90	98
·actor	250	155	128	113	88.5	89
·y, g./cc.	0.650	0.441	0.575	0.755	0.652	0.747
·ing length, in.	7100	6120	9460	14,600	12,000	13,100
·h, %	3.4	2.7	3.6	3.5	3.6	4.2

Table XIII. Comparison of Bleached Pulps

	At zero beating time			At 450 freeness		
	Cl No. 2.9	Cl No. 16.2	Cl No. 19.2	Cl No. 2.9	Cl No. 16.2	Cl No. 19.2
Freeness (Canadian), ml.	630	675	679	450	450	450
Time to freeness, min.	0	0	0	32	24	22
Burst factor	59	53.2	92	89	96	109
Tear factor	144	202	141	107	107	100
Density, g./cc.	0.700	0.544	0.726	0.765	0.706	0.789
Breaking length, in.	8700	7100	12,200	13,400	13,200	14,800
Stretch, %	3.5	3.4	3.3	3.6	3.7	3.4

·· and breaking length follow the trend which would ·expected from the bursting strength results. When ·lpulps have been refined to the desired degree, the ·ngths are well up to those of regular grades.

·ne high yield pulps are not easy to bleach and re·· large amounts of chlorine. When bleached, ·ever, their strengths are good and the yields ·h the wood are in excess of 50%. As mentioned ·re, the bleached results are included in this paper · matter of interest as there are no bleached pulps produced commercially at Bathurst. Anyone desiring to pursue the matter further would have to compare bleaching costs against wood prices to evaluate how far one should go in bleaching high chlorine number pulp.

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Bleaching Studies on Monosulphite Wheat Straw Pulp

G. H. NELSON, L. E. TALLEY, and S. I. ARONOVSKY

·at straw pulped by the monosulphite process was ·ched by single-stage and three-stage procedures. ·ous factors in the bleaching operation such as tem·ture, pH, pulp consistency, chlorine partition factor, · time of bleaching were investigated. Optimum ·ditions for single-stage bleaching were: pulp consist·· of 6 to 16%, pH of 7 to 9, and temperature of 30 to ·. Optimum conditions for three-stage bleaching ·e: use of 50 to 70% of chlorine for the first stage at ·n temperature and pulp consistency of 4 to 8%; use of · sodium sulphite and 1% sodium carbonate (basis dry ·leached pulp) at 80°C. for the alkaline extraction stage; · use of hypochlorite at a consistency of 6 to 16%, pH · to 9, and temperature of 30 to 40°C. for the third stage.

Earlier work on bleaching of straw pulps ·nonstrated the relationship between lignin content, ·ch consumption, and permanganate number for ·ps produced by the caustic soda process (*1, 2, 3*). ·vantage of three-stage over single-stage bleaching ·s indicated. Because of the high yield and relatively ·t color of unbleached monosulphite (neutral sulphite) ·aw pulp, it was thought desirable to obtain informa·n on its bleaching characteristics. This paper gives · data obtained in these preliminary bleaching studies.

EXPERIMENTAL

Combined, soft Winter wheat (Kawvale) straw ·wn in central Illinois was used for the studies. The ·ly part of the work was carried out on straw from · 1945 crop, and when the supply of this pulp was ·austed additional pulp was produced from straw of the same wheat variety grown on the same farm in 1949. The straw was chopped in a silage cutter to 1 to 3-in. length and then screened over a vibrating 40-mesh wire to remove dirt, straw dust, and fines.

The pulps were prepared by cooking the straw with 8% sodium sulphite and 3% sodium carbonate for 2 hr. at 170°C. (100 p.s.i.) in a 3-ft. rotary spherical digester which held about 60 lb. of dry straw per charge. At the end of the 2-hr. cooking period, the pressure in the digester was relieved to atmospheric, and the cooked straw was discharged into a box containing a perforated false bottom. After the straw was drained and washed with water by gravity displacement, it was defibered for about 10 min. in a laboratory 3-ft. hydrapulper, at a consistency of about 6%. The defibered material was diluted to a consistency of 0.35%, pumped over a laboratory-made riffler, and on to an 8-cut flat screen. The accepted screened pulp was used for the bleaching experiments.

The bleaching studies were carried out on 50-gram (oven-dry basis) samples of pulp. The pulps were mixed with the bleaching chemicals in a 3-qt., planetary-type food mixer equipped with a hook-stirring device operating at about 30 r.p.m. After the appropriate reaction period, the pulps were filtered, washed, treated with sulphur-dioxide water, and washed again. They were then pressed lightly, and weighed samples were removed for brightness and alpha-cellulose determinations. The remainder was dried and weighed. The yields and chemical characteristics of the pulps are given in Table I. The data for the pulp from the 1945 crop are the average for two cooks.

The various bleaching conditions studied, for obtaining a pulp brightness of about 70%, were temperature, pulp consistency, and pH, and, in the case of the three-stage procedure, partition of chlorine, and the type

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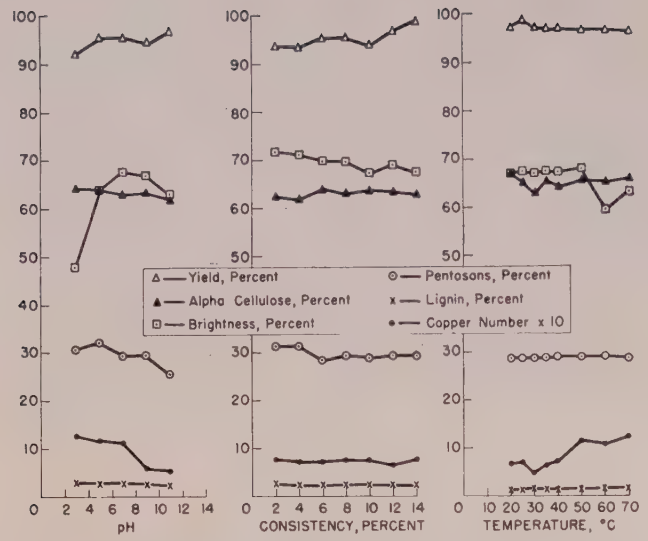


Fig. 1. Single-stage bleaching of monosulphite straw pulp—effects of pH, pulp consistency, and temperature

and degree of alkaline extraction. Attempts were made to hold constant all variables other than the one under study.

The unbleached and bleached pulp were analyzed for alpha-cellulose, pentosans, copper number, and permanganate number in accordance with the standard TAPPI procedures. Lignin and ash contents and brightness were determined by modifications of the TAPPI methods previously described (*1*, *2*). In order to determine the effects of some of these bleaching variables upon the strength characteristics of the straw pulps, a number of 2-lb. batches of pulp were bleached, and beater runs made on the bleached pulps in accordance with the standard TAPPI procedure.

SINGLE-STAGE BLEACHING

The single-stage bleaching studies were carried out on the 1949 straw. The effects of pH, consistency, and temperature are illustrated graphically in Fig. 1. As the pH was increased from 3 to 11, the brightness of the bleached pulp increased at first and then decreased, the maximum brightness being obtained at pH values of 7 to 9. The alpha-cellulose and pentosan contents and the copper number of the pulp decreased with increasing alkalinity of the bleaching solution. There was also a slight decrease in lignin content with increasing pH, but the differences were very small. The yield of bleached pulp was affected but little—the apparent increase in yield from pH 9 to 11 was due to greater difficulty in washing the pulp, as indicated by a much higher ash content.

Varying the pulp consistency from 2 to 14% in single-stage bleaching had very little effect on the alpha-cellulose or lignin content, or copper number of the bleached pulp. There was a slight tendency toward reduction of pentosans in the range of 2 to 6% pulp consistency. At the higher densities, the pentosans were unaffected. The bleached pulp yield increased somewhat and brightness tended to decrease as the consistency of pulp in the bleaching mixture was increased.

When the temperature of single-stage bleaching was increased from 20 to 70°C., the yield of bleached pulp and the lignin, pentosan, and alpha-cellulose contents were practically unaffected. The brightness of the pulp remained practically constant at bleaching temperatures up to 50°C.; beyond that point, the brightness dropped. The copper number of the pulp showed a tendency to rise with increasing temperature of bleaching.

Strength tests were run on larger-scale bleaches made at 30, 50, and 60°C., and the results are given in Table II. Single-stage bleaching did not improve the strength characteristics of the pulp, and the increase in bleaching temperature from 30 to 60°C. also had no particular effect upon the strength properties of the bleached pulps. It may be noted, however, that the time required for exhaustion of the chlorine in the single-stage bleaching tests was drastically reduced as the temperature of bleaching was increased. The exhaustion time also decreased with increasing consistency and with decreasing pH of the mixture.

It is evident from these data that the best conditions for single-stage bleaching of monosulphite straw pulp are a temperature below 50°C., a pH range of the bleaching mixture of 7 to 9, and a pulp consistency of at least 6%.

THREE-STAGE BLEACHING

First Stage—Chlorination

In the first stage, the variables studied were temperature, pulp consistency, time, and the partition factor or percentage of the total chlorine requirement for bleaching. The data are illustrated in Fig. 2. Increasing the chlorination temperature from 15 to 30°C. had no significant effects on the lignin, pentosan, or alpha-cellulose contents of the pulp. The brightness was also unaffected. There was a slight tendency toward increased yield of bleached pulp, and the copper number was increased slightly with increasing temperature of chlorination.

An increase in the chlorine partition factor also appeared to have but little effect upon the yield, constituents, or brightness of the bleached pulps, as the percentage of chlorine in the first stage was increased from 10 to 90, but there was a slight tendency toward increased pentosan content. This was probably due, as will be seen later, to the decreasing amounts of alkali and hypochlorite required. The copper number decreased somewhat as the partition factor was increased, probably for the same reason as that given for the increase in pentosan content.

Table I. Monosulphite Straw Pulps, Pulp Yield, and Chemical Pulp Constituents

Straw	*Crop year of straw used*	*Pulp yield*				*Ash in pulp, %*	*Lignin in pulp, %*	*Pentosans in pulp, %*	*Cl_2[a] used for 70 brightness, %*
		Crude, %	*Screened, %*	*Screenings, %*	*Fines, %*				
A	1945	59.8	53.6	1.9	4.3	6.8	3.9	29.0	5.9
B	1949	..	52.3	3.1	...	5.8	5.4	29.5	8.9

[a] Basis oven-dry weight of unbleached pulp.

Table II. Single-Stage Bleaching (Pulp B)—Effect of Temperature Bleaching on Strength Characteristics

Bleaching					Strength characteristics at 500 ml.—S.-R.				
Time (min.)	Temperature, °C.	Pulp consistency, %	Yield[a] of bleached pulp, %	Brightness, (Hunter), %	Beating time, min.	Bursting strength, pt./rm., lb. × 100	Tensile strength, g./rm., lb.	Tear resistance, g./rm., lb. × 100	Folding endurance, Schopper, double folds
..	..	..	..	27.1	36	76	147	43	170
97	30	12	96.4	65.8	32	80	144	41	230
20	50	12	96.9	65.1	42	81	146	40	220
15	60	12	96.8	64.8	35	74	143	43	200

[a] Basis oven-dry weight of unbleached pulp.

he variation in pulp consistency from 2 to 10%, ng chlorination, had practically no effect on the ched-pulp yield, its brightness, copper number, or in content. There was a slight increase in alpha- ulose and an approximately equivalent decrease in tosan, as the consistency was raised.

arying the time of chlorination from 10 to 60 min. practically no effect on the yield or characteristics he bleached pulp.

he effects of varying the chlorine partition factor he strength characteristics of the bleached pulp are n in Table III. At the lower partition values, the e required for exhaustion of the chlorine in the third ypochlorite stage was increased considerably. The sting and tensile strengths and tearing resistance sulphite and sodium carbonate, in amounts ranging from 1 to 4% of the oven-dry unbleached pulp. The extraction was carried out at a consistency of 12% and a temperature of 80°C. The results are illustrated in Fig. 3. The data show that the pulp extracted with a mixture of sodium sulphite and sodium carbonate was obtained in larger yield and with higher ash content than when caustic soda was used as the extractant. The sulphite-carbonate extracted material had lower lignin and pentosan contents. The larger permanganate number of the sulphite-carbonate extracted pulp is anomalous when viewed in the light of the lower lignin content of this pulp. However, too great reliance cannot be placed upon permanganate numbers in this low range.

Table III. Strength Characteristics of Bleached Pulps (Pulp A)

Bleaching[a]									Strength characteristics at 500 ml.—S.-R.				
Stage 1—Chlorination			Stage 3—Hypochlorite bleaching										
Time, min.	Pulp consistency, %	Cl_2[b] used, %	Time, min.	Pulp consistency, %	pH	Total[c] Cl_2 used, %	Yield[c] of bleached pulp, %	Brightness (Hunter), %	Beating time, min.	Burst-strength, pt./rm., lb. × 100	Tensile strength, g./rm., lb.	Tear resistance, g./rm., lb. × 100	Folding endurance, Schopper, double folds
..	..	..	..	..	..	..	..	31.2	35	96	182	52	310
60	4	26	60	12	8.1	5.1	93.0	76.2	35	100	151	61	740
30	4	48	60	12	9.0	5.2	93.3	75.0	35	96	158	56	560
30	4	80	270	12	9.0	5.4	91.5	74.6	33	101	164	56	510

[a] Stage 2—Alkaline extraction—heated at 80°C. with mixture 1% Na_2SO_3 + 1% Na_2CO_3 for 30 min. at 12% pulp consistency.
[b] Per cent of the total chlorine required.
[c] Basis oven-dry weight of unbleached pulp.

the pulps were practically unaffected as the parti- n factor was varied from 20 to 75%. The folding lurance, however, tended to increase as the partition tor was increased. It is interesting to note here that ltistage bleaching resulted in a much larger increase folding endurance of the bleached pulp than was tained in single-stage bleaching.

It is apparent from these results that the conditions chlorination or the first stage of multistage bleach- g can be varied over a fairly wide range of temperature, rtition factor, pulp consistency, and time, with no rmful effects upon the brightness, chemical charac- istics, or strength properties of the resulting pulps.

cond Stage—Alkaline Extraction

Two types of alkali were used in the extraction stage:) caustic soda, or (2) an equal mixture of sodium

As expected, the yield of pulp and its permanganate number, lignin content, and ash content decreased with the increasing amount of alkali used for extraction. A larger reduction in ash content was obtained with increasing caustic soda, but with increasing sulphite-carbonate as the extractant, the ash content of the pulp was not changed much. The type or amount of alkaline extractant had relatively little effect on the pentosan content of the pulp.

The extraction temperature was varied from 40 to 90°C., with 1% sodium sulphite and 1% sodium carbonate, and a pulp consistency of 12%. As shown in Fig. 4, increasing the temperature gave decreased lignin content and permanganate number and a slightly decreased pulp yield. The pentosan content of the pulp was practically unaffected up to a temperature of 80°C., beyond which the pentosan content dropped. The

Table IV. Strength Characteristics of Bleached Pulps (Pulp A)

Bleaching[a]									Strength characteristics at 500 ml.—S.-R.				
Stage 1—Chlorination			Stage 3—Hypochlorite bleaching										
Cl_2[b] used, %	Time, min.	Pulp consistency, %	Cl_2[b] used, %	Time, min.	Pulp consistency, %	pH	Total[c] Cl_2 used, %	Yield[c] of bleached pulp, %	Brightness (Hunter), %	Beating time, min.	Burst-ing strength, pt./rm., lb. × 100	Tensile strength, g./rm., lb.	Tear resistance, g./rm., lb. × 100	Folding endurance, Schopper, double folds
..	..	..	..	..	..	..	..	..	31.2	35	96	182	52	310
3	60	4	17	60	12	9.0	5.2	93.2	74.9	..	96	163	51	...
3	60	4	17	60	12	6.9	5.1	93.7	75.5	..	95	166	53	...
3	60	4	17	60	2	8.2	4.6	93.9	73.0	..	97	177	47	...
6	60	4	24	60	8	8.0	5.0	93.8	75.5	37	100	161	57	810
1	60	4	19	60	16	8.5	5.3	93.7	74.9	30	99	151	59	580

[a] Stage 2—Alkaline extraction—heated at 80°C. with mixture of 1% Na_2SO_3 + 1% Na_2CO_3 for 30 min. at 12% pulp consistency.
[b] Per cent of the total chlorine required.
[c] Basis oven-dry weight of unbleached pulp.

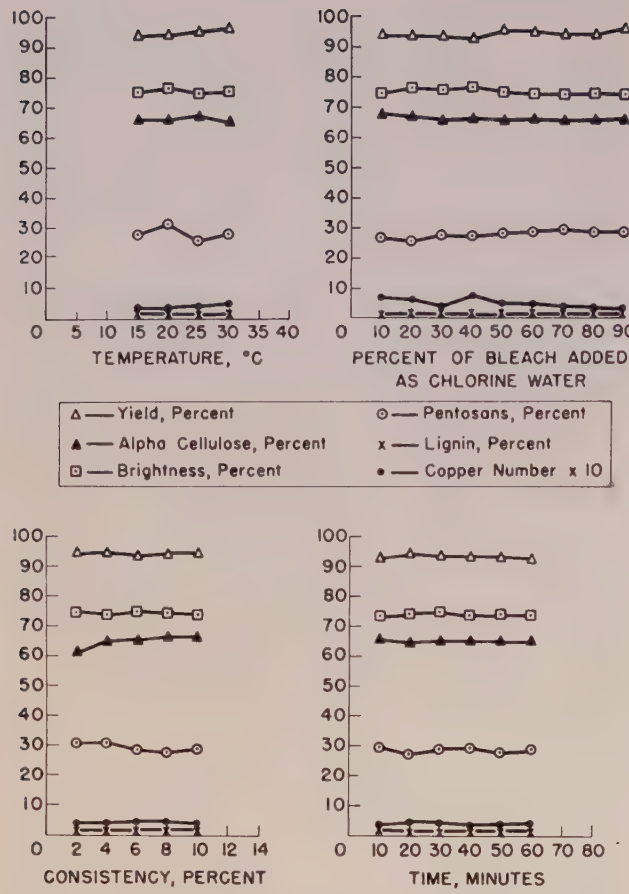


Fig. 2. Multistage bleaching of monosulphite straw pulp —effects of temperature, pulp consistency, time, and partition factor in first or chlorination stage

ash content of the pulp was unaffected by the change in extraction temperature.

It is evident from these results that the use of 2% extractant, composed of equal quantities of sodium sulphite and sodium carbonate, at 80°C. gave the optimum practical results from the standpoints of low lignin content, little or no reduction in pentosan content, and no appreciable loss in pulp yield.

Third Stage—Hypochlorite Bleach

The results from varying the temperature, pH, pulp consistency, and time in final bleaching with hypochlorite are illustrated in Fig. 5. Increasing the temperature of the hypochlorite treatment from 20 to 40°C. had practically no effect upon the pentosan, alpha-cellulose, or lignin contents or upon the copper number of the bleached pulp. An increase in brightness was noted as the bleaching temperature was raised. There was also a tendency toward higher yield with increased bleaching temperature, but the total increase in yield was fairly small.

Increasing the pH value of the bleaching mixture from 3 to 11 caused practically no change in yield or lignin content of the bleached pulp. The alpha-cellulose showed a slight increase and the pentosans a small decrease as the alkalinity of the mixture was increased. This decrease in pentosans is probably due to their greater solubility in strongly alkaline media. As noted in connection with single-stage bleaching (Fig. 1), the brightness of the bleached pulp increased at first and then decreased as the alkalinity of the bleach mixture was increased. The optimum brightness wa obtained again at pH values of 7 to 9.

Increasing the pulp consistency from 2 to 16% i hypochlorite bleaching had practically no effect on th yield or on the alpha-cellulose, pentosan, or lignin con tents of the pulp. There was a tendency toward in creased pulp brightness as the consistency of the mix ture increased. The copper number also increase slightly with increasing pulp consistency.

As in the case of consistency, increasing the time contact of the pulp with the hypochlorite bleachin mixture from 10 to 60 min. had very little effect on th yields or constituents of the bleached pulp. The brigh ness of the pulp increased as the time of bleaching in creased to 30 minutes; beyond that period the addition time did not change the brightness.

The effect of varying the pH and the pulp consistenc upon the physical strength properties of the pulp wer investigated. The results are given in Table IV. Th data show that the strength properties of the pulp wer practically the same whether the hypochlorite bleachin was carried out at pH 7 or at pH 9. Although th comparison was made with only two runs, the resu is interesting since generally it has been assumed tha

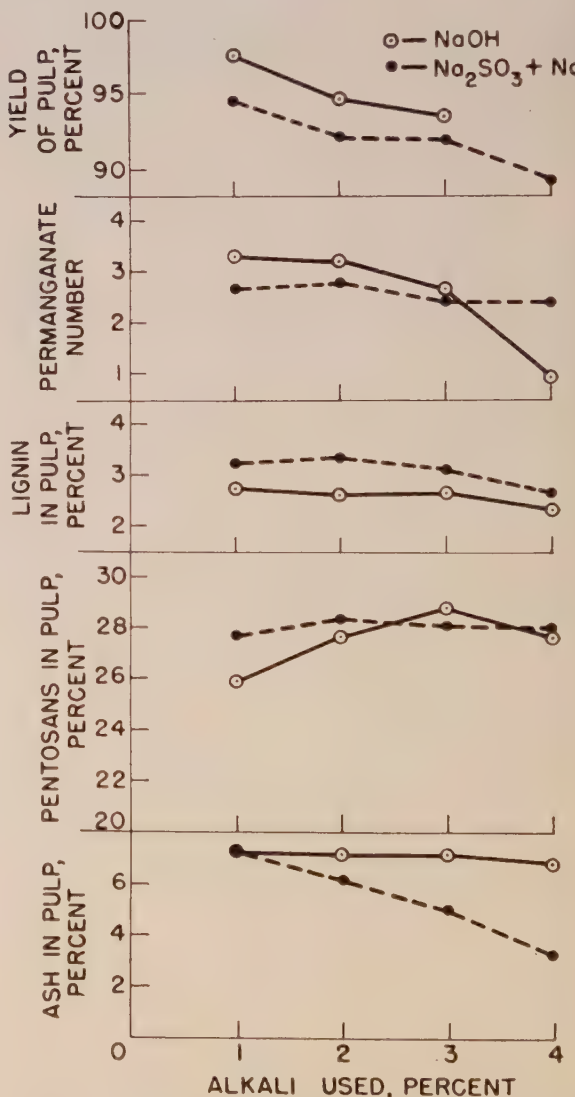


Fig. 3. Multistage bleaching of monosulphite straw pul —effects of type and concentration of alkali for extractio

ching at pH 7 would be deleterious to the strength perties of the bleached pulp.

arying the pulp consistency in hypochlorite bleach- from 2 to 16% also seemed to have but little effect n the strength properties of resulting pulps, except ibly for a slight decrease in folding endurance with easing consistency.

is therefore evident that in the third or hypochlo- stage of bleaching the brightness of monosulphite w pulp is increased with increasing temperature, istency, and time of bleaching. Optimum bright- is obtained at pH values of 7 to 9. Neither the nor pulp consistency in the hypochlorite stage had particular effect upon the strength properties of resulting bleached pulps.

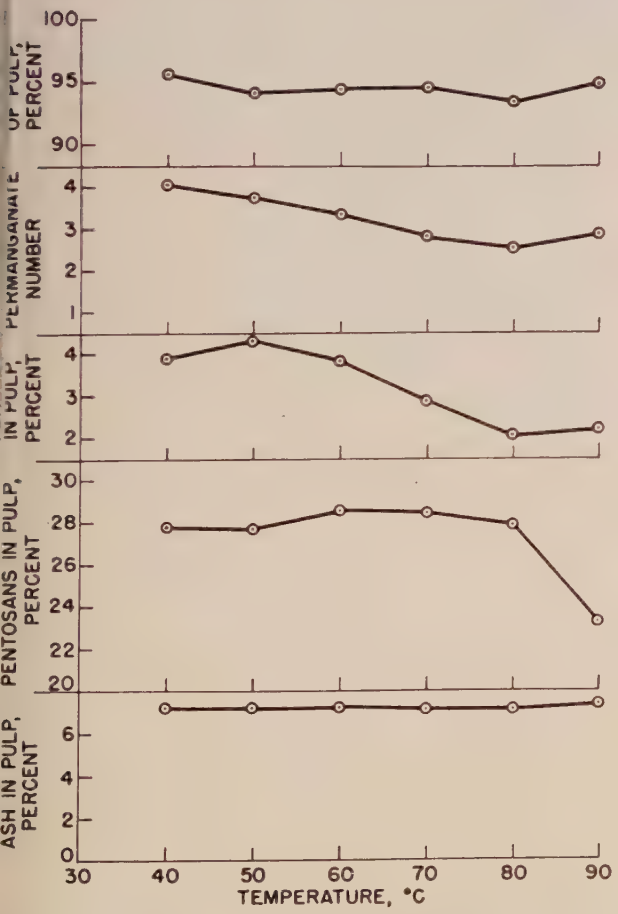
SUMMARY AND CONCLUSIONS

heat straw was cooked by the monosulphite proc- and preliminary bleaching studies were made on resulting pulps, with both the single-stage and three- e chlorine bleaching procedures. The various ors in the bleaching operations including temperature, pulp consistency, chlorine partition factor, and e of bleaching were investigated.

For optimum results in single-stage bleaching, the uired conditions were: a pulp consistency of 6 to %, a pH value of 7 to 9, and a temperature of 30 to C. Within these ranges pulps of satisfactory bright- s and good strength characteristics were obtained.

The optimum conditions for three-stage bleaching monosulphite wheat straw pulp, based on this work, are: (1) The amount of chlorine for the first or chlorination stage should be 50 to 70% of the total chlorine requirement, and the chlorination should be carried out at room temperature and at a pulp consistency of 4 to 8%. (2) For the second or alkaline-extraction stage, the pulp should be treated with a mixture of 1% sodium sulphite and 1% sodium carbonate (based on dry, unbleached pulp), for 30 to 45 min. at 80°C. The sulphite-carbonate mixture is preferable to caustic soda because the mixture gives a better yield of pulp with lower lignin content. (3) The third or hypochlorite bleaching stage is carried out by using the remainder of the required chlorine as hypochlorite, at a pulp consistency of 6 to 16%, a pH value of 7 to 9, and a temperature of 30 to 40°C.

In good agreement with experience in bleaching wood pulps, the three-stage process produced brighter pulp with better strength properties and used less chlorine than required for single-stage bleaching.



g. 4. Multisage bleaching of monosulphite straw pulp —effects of temperature of extraction

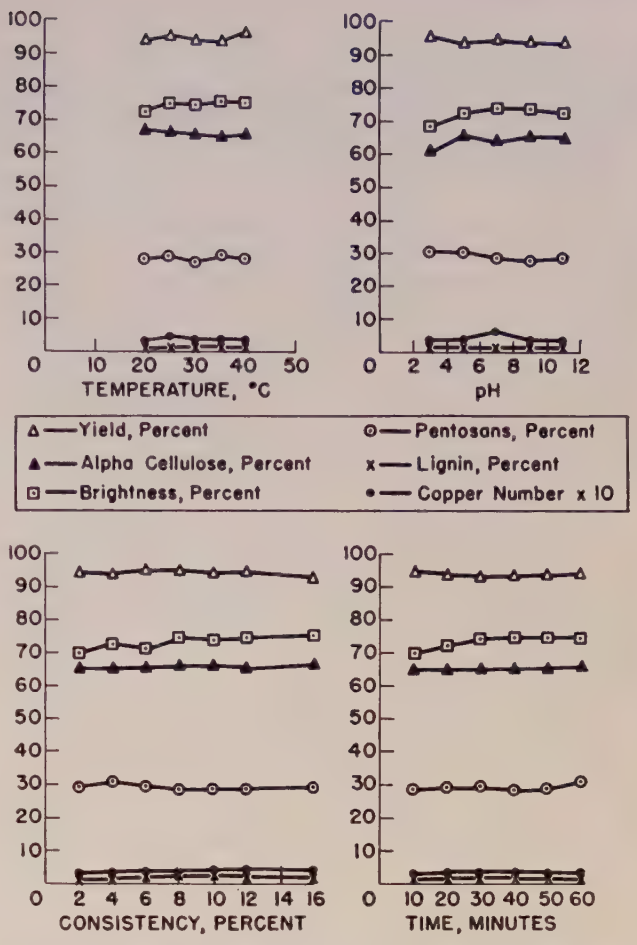


Fig. 5. Multistage bleaching of monosulphite straw pulp— effects of temperature, pH, pulp consistency, and time in the hypochlorite stage

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Organoleptic Evaluation of Paper Products for Food Packaging

L. C. CARTWRIGHT and P. H. KELLEY

An important function of packaging is to protect food products against change of aroma and flavor. While protecting products against entry of light, moisture, oxygen, and foreign odors, the packaging material itself may contribute off-odor or off-flavor to the packaged food. Organoleptic methods for evaluation of packaging material in this respect are discussed; details of procedure and examples of application to packaging problems are presented.

In the past several years, increasing emphasis has been placed on the importance of the aroma and flavor of food products. Public demand has, in many instances, forced the food manufacturer to exercise greater care and stricter control measures to insure that his product reaches the consumer without any serious deterioration in aroma and flavor. With the end of the second World War, the public began to demand better quality food products, where they had been accepting, in some instances under emergency conditions, substandard items. With constantly decreasing dollar-value in the past few years, the consuming public has been increasingly insistent upon receiving full value for each dollar. Paralleling this public demand, those in the food field have shown increasing interest in the evaluation of food products in terms of consumer acceptability, particularly as to aroma and flavor quality. Physical and chemical means for the evaluation of food palatability have been found wholly inadequate for the detection of differences in aroma and flavor which are readily apparent to the average consumer. Thus, more and more, we have been turning to organoleptic (i.e., sensory) evaluation of food products.

This specialized type of flavor evaluation has long been conducted by individual expert tasters in the wine, coffee, and tea industries. However, it takes years to train such experts, and their evaluations are almost purely subjective, with no ready means of detecting and correcting for their not infrequent serious aberrations in aroma and flavor perception and evaluation. With the need for palatability evaluation of an increasingly wide range of food products, the organoleptic panel, consisting of a number of individuals, carefully selected and trained for evaluation of a particular type of product, has come into wide use. Especially during the past ten years, there have been many contributions to the technical literature in this field. The most comprehensive survey of such literature available is found in the report of the proceedings of the national conference on palatability evaluation held in 1950 under the auspices of the Bureau of Human Nutrition and Home Economics, U. S. Department of Agriculture, Washington, D. C. (*4*).

We have used the organoleptic panel for evaluation of properties affecting sensory response for many years, and have endeavored to contribute to the improvement and refinement of the method, both as a general research tool (*2*), and as adapted to various special problems in food formulation, packaging, and other fields (*1*, *3*, *5*, *6*).

With the ever-increasing number and types of packaging materials for foods, there is a definite need for the evaluation of changes in aroma and flavor which can be traced to the packaging material itself. Such aroma and flavor changes may be due to the permeability of the material to atmospheric oxygen, moisture vapor, and/or foreign odors. Transmission of light, or lack of permeability of the packaging material to certain products formed during storage, may also cause deterioration of the food product. Pickup of odor and flavor from the packaging material, itself, is often a major contributory factor in the change of food flavors. When a new package is designed for a particular food product, all factors affecting flavor must be considered (*10*). In this paper, we consider mainly the pickup of aroma and flavor from the packaging materials, rather than permeation through the packaging material. The same general principles would apply to the evaluation of aroma and flavor changes due to other properties of the package, although preparation of the samples for aging prior to evaluation would, of necessity, be altered to suit the particular problem.

SELECTION OF SAMPLES OF PACKAGING MATERIALS

As for any test of packaging materials, it is essential that a representative sample be obtained. Sampling of several mill runs of the packaging material is generally desirable to assure that the aroma and flavor quality of the material can be maintained. Usually, the square root of the total number of units (rolls) is sampled, with the proviso that not less than five nor more than 25 units per lot are sampled. Variations in this procedure may be necessary, such as sampling at certain times of the day or from certain sections of a warehouse. It may be that there are certain outside factors which contribute off-odors to the packaging materials, and other modifications of sampling procedure may be necessary. The nature of the problem is the basis for decision as to the exact sampling procedure. Great care must be taken to see that the samples do not become contaminated during transfer from the source of sampling to the place of test. Such contamination requires taking of new samples. However, if it is not realized that contamination has occurred, erroneous results are obtained.

PREPARATION OF SAMPLES FOR ORGANOLEPTIC TESTING

Generally, as soon as a sample of packaging materia

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ceived in our laboratories, it is cut into pieces of able size to be stored in a clean, odor-free, screw- jar. The sample is thus protected from odors, and odors which the sample may have are retained in storage jar. Sufficient quantity of the sample is ed in this manner to provide uniform samples of material for all subsequent tests. An aroma eval- on of the packaging material may be all that is uired. However, in most of our work, this investi- on serves as a guide only to evaluation of off-flavor elopment which might occur in the food product. eral methods may be used for this evaluation; the ple may be evaluated by smelling a freshly torn area, smelling material that has been stored in jars for east 24 hr., or by the more rigorous tests of submer- in hot water or aging for 24 hr. at high temperature humidity. In many instances, however, we have ually tasted the material, chewing a portion for a seconds.

nsfer of Packaging Odors to a Food Product

Generally, we prefer to evaluate the actual effect of packaging material on the aroma and flavor of the rticular food product which is to be packaged. Aging packages under normal storage conditions, as they uld be distributed to the consumer, and subsequent luation of the food, would be most desirable. How- r, this is not practical because of the time necessary such tests, and the multitude of test packages which uld be required. Also, many packages are made of a mposite of various materials, and the source of an jectionable flavor could not be traced. Therefore, elerated aging tests are conducted which can be mpleted in a relatively short space of time and will ow individual components of the package to be tested. rrelation of the results of such accelerated aging ts with shelf-life tests is desirable, but may not be sential, depending upon the food product.

Whenever it is feasible, the actual food product which to be packaged is used as the absorption medium. his is not always possible and, where the particular od product cannot be used, foods are selected which ar a relationship to, and which will absorb odors as adily or more readily than, the product to be pack- ed. Aging conditions will depend upon the type of od product, and care should be taken to select a ndition which is not too extreme. Acceleration is so obtained by using a greater proportion of packag- g material per unit of food than would be found in the tual package. The packaging material can be aged contact with the food, or in the same container with- it actual contact. Generally, we prefer the former. veral methods have been reported in the literature for ansfer of odors to food products. Sandwiches of gh-score butter, or chocolate, and the packaging aterial are aged for 24 to 48 hr. in closed glass con- iner at room temperature (*8*, *9*). This allows for fferentiation of the odor properties of each side of the aterial. Small containers to hold heavy cream can prepared from the packaging material, covered, and ed for 24 hr. at 45°F. (*8*). We have also used min- al oil, weak acetic acid solutions, and distilled water similar tests. Mineral oil, placed in a dish and set ar the packaging material while it is aging, also picks odors (*8*, *9*).

In our evaluation of packaging for potato chips and flour, we have used the actual food products, aging at high humidities. Chocolate products such as candy, cocoa, and prepared cocoa mix, which we have studied in relation to their packaging materials, have shown that they are particularly susceptible to absorption of odors and flavors. For such food products, the packaging material to be tested is cut into $^1/_2$-in. squares, mixed with five times its weight of the food, placed in clean, odor-free glass jars, and stored with the cap off for a period of 7 days at 100°F., over a saturated solution of sodium tartrate which gives a relative humidity of 90%. However, reduction in temperature is required for chocolate candy. Separate portions of the chocolate product are aged alone to serve as an aged control since aging under these adverse conditions affects aroma and flavor. After the aging period, the product is separated from the packaging material and submitted to our organoleptic panel, together with aged and unaged controls. This test method can be extended to packaging materials which are not to be used for chocolate products, if the particular food bears some relationship to chocolate, and is not suitable for accelerated aging tests.

ORGANOLEPTIC EVALUATION

The general principles of organoleptic panel testing apply to the evaluation of food products aged in contact with packaging material and also to examination of the packaging material itself. The general methods have been covered in the literature and will not be repeated here. However, there are certain details which should be emphasized and, in our work, we have developed certain techniques which are particularly applicable to the evaluation of packaging material for aroma and flavor quality.

First, the system of evaluation of the packaging material must be decided upon. We suggest a combination of ratings and comments by the panel members. As stated previously, the evaluation of the packaging material before contact with the food product serves as a guide to the off-flavors which might be absorbed by the food product. In this examination, we rate the presence of odor as: (1) just detectable, (2) moderate, (3) strong, and (4) very strong, and request a description or identification of the odor. Common odors found in packaging materials have been described as: cardboard, paper, sweet, ink, glue, oil, musty, turpentine, solvent, fruity, etc. For evaluation of the food product, we generally use a system in which the presence of desirable notes in aroma, flavor, and aftertaste, and the absence of undesirable notes in aroma, flavor, and aftertaste, are scored. The total score for the unaged control sample of the food product is 100, made up of 30% for aroma, 40% for flavor, and 30% for aftertaste. Aftertaste is usually very important since off-notes encountered in foods aged with packaging material are often not apparent until the food has been swallowed or expectorated. A typical pattern of results obtained when evaluating food products by this system is given in the table below:

For this system of evaluation, the reference sample of unaged food product serves as a guide to the panel members' evaluation, eliminating preference grading. The aged control is a check on aging conditions from

one series of tests to another, since it indicates the amount of deterioration or change in the food product caused by the aging conditions and not due to the packaging material being tested.

	Unaged control	*Aged control*	*Sample aged in contact with packaging material*
Aroma			
Presence of desirable	15	13	12
Absence of undesirable	15	14	10
Flavor			
Presence of desirable	20	18	15
Absence of undesirable	20	18	12
Aftertaste			
Presence of desirable	15	13	11
Absence of undesirable	15	14	6
Totals	100	90	66

Having selected our system of evaluation, the panel members are selected. For detection of off-aromas and off-flavors, it is possible to prepare typical samples representing various degrees of contamination. Usually, we use the duo-trio technique for initial selection. The panel member must correctly select the different sample from a series of three codemarked samples in which two are alike and one is different. He must then state which sample he prefers. It is desirable that the untrained candidates' preference shall be in agreement with the predetermined standard. Further elimination is obtained by testing ability to distinguish between differing levels of the off-aroma and off-flavor.

EXAMPLES OF APPLICATION TO PACKAGING MATERIAL TESTING

Applications of these general methods have included selection of packaging material for a chocolate product and routine control testing of all mill runs of the packaging material before printing as well as after printing. In Table I, we show some test results which were obtained during our initial screening of samples. After tests of several mill runs of each type of packaging material, commercially feasible standards of acceptance were established. The requirements for component parts of the package were dependent upon the proximity of the packaging material to the chocolate product. Careful analysis of panel member scores from day to day served to check the reproducibility of their results. It was possible to reduce the number of panel members considerably without decreasing the reliability of the results by selection of the panel members showing the best record of performance. A brief statistical analysis of the total scores was made in order to determine the significance of the difference between mean values. For each mean value, the standard error was calculated as follows:

$$\text{Standard error of the mean} = \sqrt{\frac{\Sigma d^2}{n(n-1)}}$$

where

d = deviation from the mean
n = the number of scores

The difference between two mean values is considered significant if the sum of the standard errors is less than the difference between the two means.

From Table I we see that interior wrapper A, seal with glue V, and packed in boxboard B, printed wi ink W would be the most satisfactory packaging co bination. The aroma and flavor quality of the exteri wrap is not as critical as other portions of the packag Therefore, while exterior wrap C is most satisfactor A or B could be substituted. There is no significa difference between these latter two samples in aron and flavor quality.

Table I. Organoleptic Evaluation of Packaging Materi for Chocolate Products Using Ten Panel Members

Description of sample	*Total scores* Max.	Min.	Mean	S.E.M
Unprinted inside wrapper A	95	85	90.5	±1.1
Unprinted inside wrapper B	91	81	86.6	±1.7
Unprinted inside wrapper C	86	56	71.6	±3.4
Printed inside wrapper A	94	84	89.5	±1.1
Printed wrapper A, glued portion				
Glue U	92	73	81.3	±2.0
Glue V	92	81	89.1	±1.2
Unprinted boxboard A	87	70	78.9	±2.3
Unprinted boxboard B	90	81	86.1	±1.3
Unprinted boxboard C	87	79	82.9	±1.3
Printed boxboard B				
Ink W	87	79	82.9	±1.2
Ink Y	80	44	61.9	±4.2
Exterior wrap A	88	70	82.0	±2.0
Exterior wrap B	86	74	81.2	±1.7
Exterior wrap C	95	86	90.2	±1.2

The duo-trio technique of evaluation has been us in our laboratories for evaluation of foods where or small differences are apparent. Selection of packagi material for baked dinner rolls required such a pro dure. The dinner rolls were aged in contact with e of four different types of packaging materials for 48 at 90% R. H. 100°F. Each scoring session consisted three samples: one, the rolls aged alone, the seco the rolls aged with one of the packaging materials, a the third, a duplicate of one of these two samp Fifteen selections were made on each packaging ma rial. The panel members selected the sample which t believed was different and stated their prefere The results of this test are recorded in Table II. analysis of these results tables of probability are av able (7). These tables give the level of significa for a given number of correct selections. The smal the level of significance, the more probable it is that th is a real difference between the samples compa From a study of Table II, it is obvious that sample

Table II. Duo-Trio Tests for Selection of Packag Material for Dinner Rolls

Packaging material	*Correct selections, No.*	*Level of significance*	*Preferences based on correct selection, No.* Control	Packag mater
A	12	0.0001	10	2
B	8	None	4	4
C	10	0.01	7	3
D	9	0.05	6	4

very highly significantly different from the control, is not suitable for packaging the rolls. Samples C a are of intermediate quality, and sample B is most s factory in that it contributes no significant off-od off-flavor to the rolls.

The general principles outlined in this pape evaluation of the aroma of packaging materials b

ct with foods, and for transfer of odors and flavors d products, can be applied to any problem where st lies in the pickup of odors and flavors from ging materials. General organoleptic procedures e applied to evaluation of any product for any ry effect. Results of such investigations, when ated with other physical and chemical test data, d contribute to greater consumer acceptability.

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easuring the Density of Rubber-Covered Paper Mill Rolls

RALPH M. LEIGHTON

There are in use three instruments for testing er-covered rolls: Shore Type A Durometer, Adams simeter, and the Pusey & Jones Plastometer.

he accompanying set of tables compares the read- of the above three instruments for various com- ds in use at Stowe-Woodward, Inc. To give a ite comparison of these figures, they are compared tly with the values given in the Vanderbilt Hand- of 1942, page 295, "Durometer Type A versus tometer 1/8 P. at the End of a Minute."

roll covered with No. 708 compound has a reading Durometer, and a corresponding reading of 45 1/8 P. he end of a minute. This is 5 points higher than a esponding reading of 83 Durometer on the Vander- graph, which would give 40 1/8 P. Hence the 5 t difference.

is to be noted that the same compounds seem to e similar differences, but will vary greatly with dif- nt compounds. There can be as much as 19 Plas- eter points differential in a small Durometer range n theoretically there should be no difference.

here is no over-all comparison and correlation of three measuring instruments. A comparison can be made within the same stock. This can be e by plotting curves for each individual stock in use. n by this method, we can expect as high as 5 points iation between the given Durometer and Plastom- readings. This is logical since the Durometer Densimeter are spring instruments, and by their ure cannot be accurate as compared to the Plastom- .

t seems that the optimum and most accurate read- s would be the Plastometer, taken at the end of a ute exclusively, thereby eliminating the necessity comparison.

Today the Pusey & Jones Plastometer is the most ely recognized instrument employed for the density asurement of rubber-covered paper mill rolls. This rument is used by manufacturers of rubber-covered s in the United States, Canada, England, France, many, Norway, Sweden, and Finland, and many paper mills also use the instrument for checking their covers. It has become an accepted standard instrument by both the rubber roll manufacturers and the paper mills.

It is the only instrument of its type that we know of that has been adapted to cover a wide range of roll diameters and hardnesses, and has not, insofar as we know, been officially adopted by paper technical societies. In the United States the instrument is used under the ASTM designation of D 531–41.

This instrument is of the dead-weight type and is subject to certain small errors which in a practical sense are not too important. The Plastometer registers the indentation in 0.01 mm. of a steel ball 1/8 in. in diameter into the rubber under an increase in pressure of 85 to 1085 in grams, or from 3 oz. to 2 lb. 6 oz. after a period of 60 sec.

On Dec. 14, 1950, there was a meeting of the Subcommittee of the Roll Group of the Rubber Manufacturers Association, and representatives from the following companies attended:

Raybestos-Manhattan, Inc., Manhattan Rubber Div., Passaic, N. J.
American Wringer Co., Woonsocket, R. I.
Stowe-Woodward, Inc., Newton Upper Falls, Mass.
B. F. Goodrich Co., Akron, Ohio
United States Rubber Co., New York, N. Y.
Goodyear Tire & Rubber Co., Akron, Ohio
The Pusey & Jones Corp., Wilmington, Del.

This meeting was called for the purpose of standardizing Plastometers so that manufacturers and paper mills would know what tolerances existed between Plastometers and controversies over Plastometer readings could be eliminated.

Discussion showed that the difficulty was first brought to attention about a year ago when Stowe-Woodward, Inc., found they could not check the readings of a customer. This company was also aware that other Plastometers then in use were not checking properly, consequently, the reliability of the instrument was questioned.

Pusey & Jones Corp., working with the B. C. Ames Co., brought out a new type dial gage, as the difficul-

H M. Leighton, Chief Chemist, Stowe-Woodward, Inc., Newton r Falls, Mass.

ties experienced were traced to the original gages which were found to change readings with use and age.

Several Plastometers were available at this meeting, and two sets of samples were also presented.

One set of samples was presented by Stowe-Woodward, Inc., the other by Manhattan Rubber Div., and tests were run by several different operators on different Plastometers. The results showed that a Plastometer in good condition is accurate within the following tolerances, even when operated by an inexperienced, yet careful, operator:

0 to 120 Plastometer.....plus or minus 1
120 to 200 Plastometer.....plus or minus $2^1/_2$
Over 200 Plastometer.....plus or minus $7^1/_2$

In order that a clear understanding can be obtained and no confusion result, it should first be made clear that the accepted maximum tolerances long in use by the rubber roll manufactureres have not been changed—that is:

0 to 100 Plastometer.....plus or minus 5
100 to 160 Plastometer.....plus or minus 10
160 and softer.....plus or minus 15

For example, if a customer requests a Plastometer reading of 45, it is understood that the range will be from 40 to 50, with the further understanding that anything between 39 to 51 will be acceptable, to allow for the tolerances of the instrument.

The Plastometer error or tolerance would be used only when there is a question of the roll tolerance, that is, if a roll were ordered 40 to $50^1/_8$ P., and the roll manufacturer's Plastometer showed 40 to 50, while the customer's Plastometer showed 39 to 51, it would be accepted that the roll manufacturer had supplied a roll within the plus or minus 5 of the specification, using $45^1/_8$ P. as the mean point.

The instrument should be placed on a roll or sample to be tested with the four feet in firm contact with the sample and the instrument well leveled to permit perpendicular penetration of the contact point.

Contact point shall be lowered to work for three complete turns of the dial for readings up to $250^1/_8$ P. For readings over 250 and under 400, one shall make four complete turns of the dial. For readings 400 to 500, five complete turns of the dial shall be made. One will then lower the kilogram weight until there is contact and continue to lower the weight until there is $^1/_8$ to $^3/_{16}$-in. gap between the shoulder and plate. One shall count the time from the time of contact, and the reading after 60 sec. shall be considered the Plastometer reading.

GENERAL CARE OF THE INSTRUMENT

1. The instrument should be kept in the case when not in use.

2. It should be kept scrupulously clean. This is best done by wiping off the instrument with a cloth, wet with a little oil. However, under no condition should oil be put on the stem of the dial indicator. A small amount of light oil may be applied to threads on post and to sprockets. It must not be abused. If inadvertently dropped or abused, it should be returned to the Pusey & Jones Corp. for repair.

3. Pin: Only the $^1/_8$-in. pin is to be used in the Plastometer: $^1/_4$-in. pins are obsolete. (*Note:* Since

Comparison Table of Various Measuring Instruments Determining Hardnesses of Rubber Covers

Type A Durometer—Adams Densimeter—P & J $^1/_8$ in. Plastometer

Compound	*A Durometer*	*Densimeter*	*P & J 1/8-in. Plastometer Spot*	*EOM*	*Differencea*
		95 to 100 Durometer Range			
708	99	6	16	19	+13
708	96	8	19	23	+11
708	97	7	18	22	+12
708	100	4	11	17	+13
708	98	8	18	23	+15
708	96	8	20	24	+12
708	96	8	20	25	+13
204	100	6	14	18	+14
204	99	6	14	18	+12
204	100	4	12	16	+12
2319	95	9	19	22.5	+ 8.5
2319	96	8	18	22	+10
2319	99	4	14	20	+14
2319	96	8	19	22	+10
2319	97	6	20	22.5	+12.5
3272	99	6	30	25	+19
2319	96	8	21	25	+13
2319	96	8	21	25	+13
3096	95	8	21	28	+14
3096	99	7	21	27	+21
		90 to 95 Durometer Range			
708	92	12	30	33	+13
708	91	11	28	33	+11
708	93	12	26	31	+13
708	90	14	29	32	+8
708	91	13	26	31	+9
708	92	13	27	30	+10
708	94	13	26	30	+14
708	93	10	23	27	+9
708	93	10	22	26	+8
708	92	12	26	30	+10
708	94	11	26	31	+15
708	92	12	30	33	+13
708	97	9	21	25	+15
708	94	9	24	27	+11
3096	92	16	31	39	+19
3096	94	11	26	33	+17
3206	90	15	29	30	+6
2319	90	13	28	29	+5
3206	93	11	26	27.5	+9.5
3206	91	12	27	28	+6
		75 to 80 Durometer Range			
708	79	22	44	46	−4
708	79	22	46	48.5	−1.5
708	78	24	49	54	+1.5
708	79	23	47	49	−1
708	75	25	50	52	−8
708	78	24	51	53.5	+1
708	77	24	52	54	−1
708	76	24	54	56.5	−1
708	78	24	52	54	+1.5
3096	78	24	48	51	+1.5
3096	75	25	53	57	−3
3096	79	22	53	58	+8
3096	75	24	48	49	−11
3272	78	23	46	48	−45
3045	78	23	47	50	−2.5
3060	75	25	54	56	−4
5060	75	24	56	58	−2
5060	78	21	50	51	−1.5
5060	79	22	48	50	0.0
5060	77	22	48	50.5	−4.5
5060	78	22	47	49	−3.5
		70 to 75 Durometer Range			
708	74	27	56	58	−4.5
708	74	26	58	60	−2.5
708	73	27	59	61.5	−3.5
708	72	30	63	64	−3.5
708	70	30	65	66.5	−6
708	73	29	60	61	−4
708	71	41	65	67	−3
708	73	30	61	63	−2
3096	73	27	59	61	−4
3096	72	29	61	64	−3.5
3096	71	26	56	58	−12

parison Table of Various Measuring Instruments in ermining Hardnesses of Rubber Covers (*continued*)

Com-pound	A Durom-eter	Den-simeter	P & J 1/8-in. Plastometer Spot	P & J 1/8-in. Plastometer EOM	Difference[a]
5403	73	26	63	67	+2
5403	72	27	64	69	+1.5
5060	72	27	65	67	−0.5
5060	71	27	62	65	−5
		35 to 40 Durometer Range			
300T	35	79	180	187	−8
300T	37	71	173	178	−7
300T	37	77	171	174	−11
300T	37	75	180	185	0.0
300T	39	75	179	182	+6
300T	36	80	190	196	+6
2381	37	80	191	196	+11
300T	40	85	212	221	+49
5383	38	75	200	214	+24
5383	38	75	208	223	+43
2381	37	78	198	201	+16
1932	35	82	214	224	+29
1932	36	82	205	219	+29
2381	37	80	211	214	+29
3304	38	75	120	178	−2
		30 to 35 Durometer Range			
300T	34	82	196	200	0.0
300T	34	88	195	199	− 1.0
300T	33	83	190	197	−10
300T	30	97	252	267	+40
2381	32	90	217	225	+11
2381	32	92	235	248	+34
2381	34	83	218	221	+21
2381	31	96	244	250	+30
2381	32	93	250	254	+40
2381	33	88	223	228	+21

Difference in Plastometer readings from base curve. Base figures are ed from type A Durometer versus P & J 1/8-in. Plastometer EOM. ersion chart based on graph, page 295, of Vanderbilt Rubber Handbook

re have been recent changes in the 1/8-in. Plastom- pin, one should see that there are no old spare s available in plant or laboratory, because pins more n 2 years old are not of the same contour as the sent pin and will not give the same reading. All h old pins should be discarded.)

. Pin Stem: The rod connecting the pin with the icator. It is most important that this stem be kept e. A bent stem will give erroneous results. The eness of the stem can easily be determined by rolling n a flat plate. If it is bent it will kick up. It is st important that the plate supporting the kilogram ght and the base plate of the instrument be parallel. hey are not, there will be a tendency for the instru- nt to kick up and read incorrectly.

5. The four feet of the instrument should not be owed to wear to the point where the fit is too sloppy, herwise it will be difficult to set the instrument on a l to get a good reading.

6. If the user has a Plastometer and wishes to check instrument to see if it is reading correctly, it is sug- sted he do as follows:

a) Obtain an extra Ames 214 dial gage indicator with post ned to gage. This should preferably be obtained through Pusey & Jones Corp. The dial indicator from the question- e Plastometer is then removed and the new dial indicator with post is substituted. If the questionable Plastometer then cks with the new dial gage one can assume that the remainder he Plastometer is in good order and is reading correctly.

b) If, after the dial indicator has been changed and the stometer still does not check, one can assume that other parts he Plastometer are not working correctly, and the instrument uld be returned to Pusey & Jones for a check.

c) When a dial indicator is substituted care must be exercised ttaching to the Plastometer so that the dial spindle and rod are tered with the hole in the top of the weighted extension. If new dial is satisfactory and is to be attached permanently to the Plastometer, this proper position on the post should be marked, using a pencil through the set screw hole in the bottom plate. The post is then countersunk, using the tip of a drill to receive the set screw so that it will keep the dial indicator permanently in the proper position. If the gage is to be used for checking only, the post should not be drilled, so that it can be accommodated to any Plastometer.

Durometer Type A versus Plastometer P & J 1/8-In. P. EOM Conversion Chart

Based on Graph on Page 295 of Vanderbilt 1942 Rubber Handbook

Durometer	EOM	Durometer	EOM
100	4	63	91
99	6	62	94
98 Esti-	8	61	97
97 mated	10	60	100
96	12	59	103
95	14	58	106
94	16	57	110
93	18	56	114
92	20	55	117
91	22	54	120
90	24	53	123
89	26	52	126
88	28	51	130
87	30	50	134
86	32	49	137
85	35	48	140
84	38	47	144
83	40	46	148
82	42.5	45	152
81	45	44	156
80	47.5	43	160
79	50	42	164
78	52.5	41	168
77	55	40	172
76	57.5	39	176
75	60	38	180
74	62.5	37	185
73	65	36	190
72	67.5	35	195
71	70	34	200
70	72.5	33	207
69	75	32	214
68	77.5	31	220
67	80	30	227
66	83	29	234
65	86	28	240
64	89		

The Pusey & Jones Corp. is undertaking to notify all users of Plastometers on record who have not returned their instruments within the past 6 months for a check, of this change of dial gage indicator.

It is important that this be done because the old indicating gages will not in general check with the new dial gage now being put on all new Plastometers and all repaired Plastometers.

Pusey & Jones will be glad to advise any user of the Plastometer on any apparent difficulties, or where there seems to be a nonuniformity.

From the above information, any laboratory can check the minor difficulties usually encountered with a Plastometer, and any of the rubber roll covering manufacturers would be glad to check a Plastometer, either in person or by sending a check sample to them.

It is not at all difficult to check the various small parts of the Plastometer to see that they are accurate and in good working order, and if the above tolerances are adopted, it is believed that there should be no difficulty in the paper mills checking their specifications on rolls as furnished by the rubber roll covering manufacturers.

Note: This paper was written for the old type Plastometer. Pusey & Jones has since developed a new model. However, the results obtained are the same with both instruments.

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An Investigation of the Homogeneity of Isolated Native Black Spruce Lignin

CECIL L. HESS

A sample of isolated native black spruce lignin was prepared which was similar to that prepared by Brauns with regard to the methoxyl content but had a higher carbon content. This material was separated into a series of fractions by fractional precipitation with dioxane as the solvent and benzene as the precipitant. Precautions were employed to obtain the fractions from a system in equilibrium. By this method a total of 54 fractions was obtained which were combined to give eleven composite fractions. The initial fractions were difficultly soluble and of a darker color as compared with the final fractions, which were quite soluble and of a light bright color. Molecular weights of the fractions were obtained from osmotic pressure measurements. The native black spruce lignin was composed of constituents having molecular sizes from 2800 to 6700. It is probable that the range extended both higher and lower. About one third of the material had a molecular weight of 4500 to 5000. Twenty per cent of the material had a molecular weight higher than this value and the remainder was lower. A tentative relationship was also found between the solution viscosity of the fractions and their molecular weights. Heating lignin in either dioxane or methyl cellosolve solutions caused a small decrease in the solution viscosity. A small decrease in the molecular weight was found, as well as some change in the ultraviolet spectrum of the unfractionated lignin. These changes were concluded to be of a minor nature as far as the heterogeneity of isolated native lignin was concerned. The methoxyl contents and the carbon and hydrogen values for the fractions differed from those of the unfractionated lignin. Neither were the ultraviolet visible nor infrared spectra of the fractions identical. Differences in the infrared spectra of the fractions occurred primarily in the carbonyl region. It was concluded that isolated native black spruce lignin was a mixture of substances which differed both in their molecular structure and in their chemical constitution.

Brauns (*1*) extracted isolated native lignin from black spruce sawdust under conditions designed to prevent any changes in the lignin. Products prepared by Brauns from the isolated native lignin were similar to the corresponding products prepared in an almost quantitative yield directly from the wood. Hence, this lignin appears to be a desirable material for further investigations of lignin chemistry.

Investigations have shown that both natural occurring polymers and synthetic polymers—e.g., cellulose, proteins, polystyrene, and others—are mixtures (*2*). Many natural polymeric materials are known; yet none of these is known in its pure form. They may differ in molecular size, in molecular shape, or in chemical composition. These variations will have a marked influence on the properties of the material. In considering any material, it is desirable to know to wh extent that material is homogeneous or a mixture. the material is not homogeneous, the differences amo the constituents become of interest.

The physical properties of various lignin preparatio have been investigated to some degree. Among th properties which have been investigated is the hom geneity of certain lignin preparations. Staudinge and Dreher (*3*) fractionated a formic acid spru lignin from dioxane with water. They were able separate the lignin into four fractions which differe in their solubilities and in their methoxyl conten Only small variations were indicated by the viscosit measurements. The molecular weight measuremen showed no differences between the various fractions.

Patterson and co-workers (*4*) have fractionated crude maple ethanol lignin into a series of fractio according to their solubilities in different solvent The fractions obtained differed with regard to the reduced viscosities, elementary analyses, and methoxy contents. Refractionation of the various solubiliti groups leads to the conclusion that each solubiliti group was a heterogeneous system.

Bailey (*5*) has examined a series of lignins isolat by extracting wood with butanol at 160°C. in the pre ence of hydrochloric acid. He washed his extrac with water and dried the residue. These produc were then examined by a method of paper chromato raphy, using butyl acetate as the developer and 1,4 dioxane as the solvent. The solution was applied to th center of a filter paper and allowed to diffuse outwar toward the edge. It is not surprising that he foun marked and varying heterogeneity among preparatio from different species of wood because the preparatio were not rigorously purified.

Stumpf and Freudenberg (*6*) subjected lignin fro spruce and beech wood meal to a fractional extractio The wood meal was swollen with water and the wate was partially displaced with moist dioxane containin 10 to 20% of water. Dioxane containing 1% of wate and 0.1 *N* hydrochloric acid was used to extract th lignin. Initially, a hydrogen chloride-free ligni solution was obtained. About 85% of the lignin wa extracted from beech and a little less from spruce The first 5 to 7% agreed in its composition with th isolated native lignin as obtained by Brauns (*1*) Although the elementary composition remained pra tically constant, the methoxyl content was found t rise slowly and become constant at 16% after abou one half the lignin had been extracted. The ultra violet and infrared spectra of the individual fractio were very similar.

Lovell and Hibbert (*7*), by means of a fractiona

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ibution system, separated an isolated native n lignin into three fractions. These fractions were d to differ in their methoxyl contents and their ion viscosities. E. J. Jones (*8*, *9*) also fractionated nple of isolated native black spruce lignin from a ane solution with benzene and petroleum ether. d the infrared spectra of these fractions were ex- ed. The main differences in the fractions were nd in the carbonyl region.

ne molecular weight of a material is also an im- ant property in connection with structural studies. of further importance for a full understanding of the re of a material. The molecular weight of lignin been determined by a variety of methods. Molec- r weights varying from 250 to 11,000 have been rted (*10*). This variation in the molecular weight aused, most certainly, by true differences among lignins but, in addition, by the different molecular- ght averages obtained by the various methods. he molecular weight is determined by cryoscopic, ebullioscopic, osmotic pressure, or isothermal distil- on methods, a number-average molecular weight btained. If the molecular weight is determined by mentation, diffusion velocity, or free diffusion hods, a weight-average molecular weight is ob- ed. These two averages will be the same only for stem having molecules of the same size. Further- e, attention should be called to the fact that free usion is a function of the volume and the shape of particle rather than its mass. Solvation is another or to be considered in connection with measure- ts of the molecular weight by diffusion.

ramec and Pirkmaier (*11*) determined the molecular ght of an alkali hydrochloric acid spruce lignin, solved in ammonium hydroxide, by the osmotic ssure method and found it to be around 4000. ughborough and Stamm (*12*) measured the molec- r weight of various maple lignin preparations by the llioscopic and the osmotic pressure methods. They nd a molecular weight of approximately 3900 by h methods. By a diffusion method, they obtained olecular weight of 10,000. Freudenberg and Heim- ger (*13*) have measured the molecular weight of a nthetic lignin which is a dehydropolymerizate of niferyl alcohol by an isothermal distillation pro- dure described by Barger (*14*) and have found a lecular weight of 820. For the methylated syn- tic lignin and for a methylated lignin extracted m methylated sprucewood with formic acid in the sence of acetyl chloride, molecular weights of about 00 were obtained by Freudenberg and Kraft (*15*).

E. J. Jones (*16*) measured the diffusion weight for lated native black spruce lignin using the Northrup ous plate diffusion cell and quercitin as the reference terial for the determination of the cell constant. A ue of 1450 was obtained using the cube root relation- p $D\sqrt[3]{M} = K$ based upon the Sutherland-Einstein uation.

Only a limited number of investigations have been de concerning the physical properties of isolated ive lignin. In addition to those mentioned above, ading (*17*) investigated the ultraviolet spectrum of lated native black spruce lignin. E. J. Jones (*9*) mined the infrared spectrum of both isolated native ck spruce and isolated native aspen lignin. G. Jones (*18*) obtained the visible spectrum of a bleached and an unbleached isolated native lignin from eastern spruce groundwood.

To add to the knowledge concerning the physical properties of isolated native lignin, a study of the homogeneity of isolated native black spruce lignin was undertaken. The lignin was first separated into a series of fractions and their physical and chemical nature were then studied to determine what differences, if any, are to be found among the fractions.

FRACTIONATION

To ascertain the homogeneity of isolated native lignin, an attempt was made to separate the isolated material into its components. Although the method of separation selected is of the utmost importance for obtaining a resolution of the components, there is no rule to indicate the most suitable method to be used; therefore, consideration was given to the mechanics of the process. A fractional precipitation procedure seemed to be the most suitable method for an initial attempt to separate isolated native lignin into its constituents. Problems of manipulation appeared to be less and a large number of fractions appeared to be possible. It has been found that, if an equilibrium is maintained as the turbidity develops, a better resolution is obtained (*2*, *19*). This may be done by warming the turbid solution until the turbidity clears and allowing the system to cool slowly. A series of preliminary experiments was made to determine the ability of solvents to retain isolated native lignin in solutions and of nonsolvents to throw it out of solution. The solvents used were dioxane and methyl cellosolve. The precipitants used were ethyl ether, benzene, anisole, low-boiling petroleum ether, high-boiling petroleum ether, hexane, heptane, and water. The precipitant was added dropwise to 5 ml. of a 1% solution of lignin which was agitated by shaking. The end point desired was such that the turbidity developed could be cleared by warming the mixture to about 50°C. These results are tabulated in Table I.

Water was found to be the poorest precipitant. In addition to requiring a larger volume to produce a turbidity, the resulting precipitate was colloidal and did not settle freely. Heptane was found to require the smallest volume to produce a turbidity. These data indicate that methyl cellosolve is a better solvent than dioxane since it required larger concentrations of the precipitant to produce a turbidity. The system selected for the fractionation was dioxane as the solvent and benzene as the precipitant. [This system was

Table I. Solubility Relationships of Native Lignin
Ratio of the Volume of Precipitant to the Volume of Solution

	Solvent	
Precipitant	*Dioxane*[a]	*Methyl cellosolve*[b]
Water	1.08	..
Anisole	0.666	3.72
Benzene	0.377	3.19
Ethyl ether	0.278	1.29
Petroleum ether		
b. 65–110°C.	0.186	0.877
b. 30–60°C.	0.158	0.606
Hexane	0.142	..
Heptane	0.137	..

[a] Concentration—1.012 g./100 ml.
[b] Concentration—1.010 g./100 ml.

similar to that used by Jones (*9*), except that he also used petroleum ether in addition to benzene as a precipitant.] Benzene has a boiling point below that of dioxane so that no difficulty was encountered in concentrating the fractionation solution. Also, the boiling point of benzene was sufficiently high to avoid an excessive loss of benzene when the turbid, fractionating system was warmed to clear the turbidity.

With the above background, a fractionation of isolated native lignin was undertaken employing conditions which were designed to produce each fraction from a system in a state of equilibrium. A solution of 2.01 grams of lignin in 100 ml. of purified dioxane was prepared. This solution was placed in a 50-ml. centrifuge bottle and allowed to come to temperature equilibrium in a water bath controlled at 25.0°C. Benzene at room temperature (approximately 25°C.) was then added slowly to the agitated solution until a turbidity developed. The degree of turbidity was observed against a white enamel background broken by a black stripe. The mixture was then warmed in a beaker of water and stirred until the turbidity cleared. With a little experience, it was possible to develop a turbidity which could be cleared at 40 to 50°C. After the turbidity had cleared, the stirrer was removed from the mixture, the bottle was stoppered, and the system (including the heating bath) was allowed to cool to room temperature. The solution was transferred to a constant temperature bath and was allowed to stand from 8 to 24 hr.; the longer period was finally selected as the standard time. Following a short centrifuging, the clear solution was decanted into another centrifuge bottle and placed in the constant temperature bath preparatory to taking the next fraction. The fraction which had been separated was washed with about 10 ml. of solvent having the same ratio of precipitant to solvent as the solution from which it was separated. The precipitate was redissolved in dioxane, precipitated into absolute ether (in a small, conical-shaped centrifuge bottle suitable for weighing), washed with ether, benzene, and petroleum ether, dried, and weighed. A total of 54 fractions was obtained. Fifty fractions fell into the regular sequence of the fractionation. The fifty-first fraction was obtained by cooling the system to 10°C. and the fifty-second fraction was the recovered residual material from the fractionation. The last two fractions did not fall into the regular sequence of the fractionation. They include the washings from all the fractions and, therefore, were a heterogeneous mixture of all the fractions.

Table II. Combined Fractions

Combined fraction	*Fractionation*	*Fraction*	*Part of the total substance, %*
A	I	1–4	0– 11.4
A-6	II	6	5.6– 7.8
B	I	5–9	11.4– 20.0
C	I	10–13	20.0– 29.0
D	I	14–16	29.0– 36.5
E	I	17–21	36.5– 43.6
F	I	22–30	43.6– 51.7
G	I	32–37	51.7– 59.0
H	I	38–45	59.0– 66.2
I	I	46–52	66.2– 73.4
J	I	Washings I[a]	73.4– 94.5
K	I	Washings II[b]	94.5–102.0[c]

[a] Recovered by concentration and reprecipitation of the wash liquor.
[b] Recovered by evaporation of the final wash liquor.
[c] This value exceeds 100% because the washings from the combined fractions were added to the washings from the initial fractions.

These two fractions were the ether-insoluble and th ether-soluble washings. In order to have enough ma terial for study, it was necessary to combine the fra tions. Sufficient of the separate fractions were dis solved and reprecipitated to obtain a gross fractio of about 150 mg. The wash liquors were saved an recovered. A recovery of 102% was obtained from th entire fractionation. This was high because the wash ings from the combined fractions were added to tho of the initial fractions.

Because of the volume of the fractionating solution it was necessary to concentrate the system after th twenty-first and the forty-third fractions. The firs concentration was effected by a dropwise evaporatio *in vacuo*. The second concentration and the fina concentration were accomplished by freezing th system and subliming the solvents. This latter pro cedure was preferred since there was no danger of over heating the solution. The recovered lignin was mad up to approximately a 2% solution and the fractiona tion was continued.

A second fractionation of the isolated native ligni was initiated to obtain some of the initial fraction which were lost from the first fractionation. Thi fractionation was carried out as before, except tha the fraction which was obtained was washed with abou 10 ml. of fresh solvent having the same compositio as the system from which it was separated. Thes washings were returned to the fractionation system The fraction was then dissolved immediately, re precipitated into anhydrous ethyl ether, washed wit benzene and low boiling petroleum ether, dried, an weighed. Because a larger initial sample was em ployed, the fractions were large enough so that it wa not necessary to combine the fractions.

The combined fractions are enumerated in Table II From this list of fractions, a selection was made fo evaluation. This will be discussed in later section Fraction A-6 is the first fraction from the secon experiment completely soluble in dioxane without th aid of alcohol. Fraction J was obtained by concentra ing the wash liquors and reprecipitating them in th usual fashion. Fraction K represents that portion o the washings which did not precipitate out with fractio J—i.e., the ether-soluble portion of the washings.

In general the primary purpose of the fractionatio was to separate the initial material into more homo geneous fractions which were then studied. However a few observations concerning the nature of the iso lated native lignin were made from the fractionatio experiments. The initial fractions were very difficultly soluble in dioxane; in fact, a portion of these fraction was insoluble even when a trace of alcohol was added (Alcohol was necessary to obtain a solution of the firs 4 to 5% of the total material separated.) In contrast as the fractionation was continued, the solubility of th fractions seemed to increase to such an extent that th recovery of the last few fractions became very difficult even on a semiquantitative basis. After forty thre fractions had been separated, 0.303 gram of materia remained. The fractionation was continued in orde to separate this into two fractions of 0.150 gram each By following the same procedure as for the previou fractions, nine additional fractions were separate whose total weight equaled 0.170 gram. This was

very of 56.1% for this stage of the fractionation compared with a recovery of 76.6% obtained for the vious 43 fractions. The decreased recovery indi- d an increased solubility of the later fractions.

the isolated state, the initial fractions were darker d the original isolated native lignin or the final ions; the initial fractions were a dull tan color; fractions were a bright, light straw color; and final fraction was a bright yellow orange. The bility characteristics and the appearance of the ions suggested that isolated native lignin was a rogeneous material.

MOLECULAR WEIGHT

Nearly all polymeric materials are heterogeneous respect to the molecular size of their components. his respect, it was of interest to study first the ecular size of the various fractions. After a con- ration of the methods which might be used to sure the molecular weight, the osmotic pressure hod was selected. Though the molecular weights e measured were at the lower limit for which osmotic ssure measurements are considered to be applicable, ecular weights for lignin preparations (*12*), as well or other materials having molecular weights in this on (*20*), have been measured successfully by this hod. A characteristic of osmotic pressure meas- ments which makes this method especially useful is t small molecular impurities, such as water and low lecular weight organic constituents, may equilibrate ough the membrane and thereby do not affect the lts of the measurements. As a result of these con- rations, the molecular weights of the fractions e determined by means of osmotic pressure measure- nts with a Zymm-Myerson type osmometer (*21*). e osmometer membranes were prepared from 600- e cellophane swollen in 10% potassium hydroxide $^1/_2$ hr.

Before the molecular weight measurements were gun, the membranes were conditioned with several in solutions to permit the absorption of the lignin the membranes to come to an equilibrium. This sorption appeared to be an irreversible process and l not affect subsequent osmotic pressure measure- nts. All the membranes prepared were found to rmit some diffusion of the solute through the mem- ane. This is a common occurrence characteristic osmotic pressure measurements made with solutes ving molecular weights below 10,000. When diffu- n does occur, the observed pressure approaches an uilibrium value, after which it drops at a constant e. To correct for this, the constant rate line is trapolated to zero time and this value is taken as the motic pressure of the solution. This is a common ocedure applied under these conditions (*20*).

The osmotic pressure for three different solutions of ch fraction was determined. The reduced pressure s calculated and plotted versus the concentration. rough these points the best straight line having the me slope as that of the unfractionated lignin was awn. The molecular weight of the unfractionated nin, as well as the slope of the pressure-concentration ationship, was obtained from measurements on nine ferent solutions. When these data were averaged the method of least squares, the slope was found to be y nearly a straight line. The osmotic pressure data and the molecular weights calculated therefrom are tabulated in Table III.

The unfractionated isolated native spruce lignin had a number-average molecular weight of 4200. (This value is in the same range as those determined for a variety of maple lignins by Loughborough and Stamm who found the number-average molecular weight of their preparations to be 3900.) The molecular weights found for the fractions extended from 2800 to 6700. Thirty-two per cent of the material had a molecular weight of 4500 to 5000; 15% had a molecular weight from 5000 to 6700, and 5% *may* have had a molecular weight above 6700, judging by its lack of complete solubility in dioxane; 22% had molecular weights from 2800 to 4100. The remaining material (26% of the total) was obtained from the washings for each fraction. This material was separated into an ether-soluble and an ether-insoluble fraction. The ether-insoluble fraction, equal to 21% of the total material, had a molecular weight of 3400. It has already been

Table III. Osmotic Pressure Data for Isolated Native Black Spruce Lignin and Its Fractions

Fraction	c^a, *g./100 ml.*	π^b, *cm.*	π/c
Unfractionated	0.0774	4.98	64.3
	0.1090	6.37	58.5
	0.1268	7.05	55.6
	0.1468	8.15	55.5
	0.1468	8.15	55.6
	0.1468	8.36	57.0
	0.1595	9.79	61.4
	0.1773	10.75	60.6
	0.1984	12.45	62.7
	Intercept		58.7
	M_n		4200
	Range		600
A-6	0.1304	4.33	33.7
	0.1738	6.62	38.1
	0.1978	7.60	38.4
	Intercept		36.7
	M_n		6700
	Range		900
C	0.1275	7.07	56.5
	0.1590	7.85	49.4
	0.1912	9.42	49.3
	Intercept		51.7
	M_n		4700
	Range		700
E	0.1281	6.97	54.4
	0.1483	8.19	55.2
	0.1669	9.17	55.0
	Intercept		54.9
	M_n		4500
	Range		100
F	0.1225	6.52	53.2
	0.1453	7.30	50.2
	0.1609	8.14	50.7
	Intercept		51.4
	M_n		4800
	Range		300
G	0.1084	6.54	60.4
	0.1302	8.13	62.4
	0.1504	8.68	57.6
	Intercept		60.1
	M_n		4100
	Range		300
I	0.1203	10.44	86.7
	0.1397	12.46	89.8
	0.1598	13.08	81.9
	Intercept		86.1
	M_n		2800
	Range		300
J	0.0573	4.63	80.8
	0.0892	5.28	59.2
	0.1099	8.32	76.0
	Intercept		72.0
	M_n		3400
	Range		1100

[a] Concentration in grams per 100 ml.
[b] Centimeters of dioxane, extrapolated to zero time.

Table IV. Viscosity-Molecular Weight Relationships for Isolated Native Black Spruce Lignin

Sample	η	M_n
Unfractionated	0.0562	4200
F	0.0775	4800
I	0.0420	2800

pointed out that a difference in solubility of the initial and the final fractions was observed. The fact that the molecular weight of this fraction was below that of the initial material indicated that this fraction contained a greater concentration of the lower molecular weight homologs than were present in the unfractionated lignin. These results led to the conclusion that the isolated native spruce lignin contains an appreciable amount of material having essentially the same molecular weight—namely, 4500 to 5000—together with both smaller and larger constituents.

Viscosity Data

Viscosity measurements on dilute polymer solutions offer a rapid method to obtain the molecular weight of polymers. Consequently, a limited number of viscosity measurements were made to determine the relationship between the solution viscosity and the molecular weight. The intrinsic viscosity $\left(\lim_{c \to 0} \eta_{sp}/c\right)$ of dioxane solutions was determined for the unfractionated material and for two of the fractions. These results are tabulated in Table IV. These data indicate that a relationship exists between the solution viscosity of lignin solutions and the molecular weight. From these data, it is possible to calculate the constants for either the original Staudinger equation or the modified equation. These relationships, however, are only tentative because of the narrow range of molecular weights investigated, as well as the small number of measurements made.

During the course of the investigation, the question of the stability of the isolated native lignin was considered and preliminary experiments were carried out on the heat stability of lignin. When solutions of lignin were heated, a decrease in the viscosity of the solution was observed which increased as the period of heating was extended. Moreover, the effect of heating increased as the temperature was increased. Temperatures ranging from 10 to 50°C. were investigated. For a 2% solution of lignin in dioxane, heated at 50°C. for 110 hr., the viscosity change (reduced viscosity which is the specific viscosity per unit concentration) was from 0.0666 to 0.624. The molecular weight of the heated material decreased from 4200 to 3500, a decrease of 700. Several possibilities may have contributed to the cause of this effect. On the one hand, it is quite possible that lignin may be associated in solution, in which case heating the lignin solutions should produce a dissociation. On the other hand, association is usually a reversible process, because of which there should be no observed change when the measurements are made at the same temperature—25.0°C. Because a change was observed in the viscosity measurements made at the same temperature, the change would appear to be an irreversible one. When a heated and an unheated solution of lignin were recovered by precipitation, a similar difference in their viscosities was found to exist before and after the recovery. Consequently, it would seem that heating the lignin solution may have caused a change in the lignin molecule. If such were the case, the decreased viscosity would indicate that the change in solution was a degradation. Even if this is the case, the change produced by heating a solution should not significantly affect the results of the fractionation. For example, two fractions having molecular weights of 5800 and 3100 had reduced viscosities for 1% solutions of 0.0979 and 0.0554, respectively; heating a lignin solution at 50°C. for 110 hr. caused the reduced viscosity for a 1% solution to decrease from 0.0666 to 0.0624. It would seem, therefore, that heating the lignin during the fractionation did not produce the variation in the molecular size of the lignin fractions. Recognizing that heating lignin in solution to 50°C. has a small effect, it was concluded that the molecular weights found for the fractions were representative of the isolated native lignin and that the heterogeneity was not produced by the fractionation.

COMPOSITION

Methoxyl Content

In examining the nature of the fractions, it was of further interest to investigate their homogeneity from a chemical standpoint. A criterion of purity, often applied to lignin preparations, is a constant methoxyl content. The methoxyl contents of the fractions are tabulated in Table V. They were not the same as that of the unfractionated material which was 15.1%. The higher molecular weight material had a methoxyl of 15.5 to 15.7%. The residual fraction had a methoxyl of 14.1%. All the other fractions, representing 74% of the total material by weight, had a methoxyl of 14.9% or above. About 50% of the isolated native lignin had a methoxyl content of 15.5%. The high molecular weight fractions tended to be rich in methoxyl and the low molecular weight fractions poor in methoxyl. The differences observed in the methoxyl contents of the fractions suggested that isolated native lignin was not only heterogeneous with respect to the molecular size of its constituents, but also with respect to the chemical constitution of its constituents.

Elementary Analysis

Carbon and hydrogen contents were determined for the unfractionated lignin and several of the fractions. These results (Table VI) indicate that the middle fractions have higher carbon contents than the initial or final fractions. Fraction J, having a low molecular weight, had the lowest carbon content.

The chemical analyses furnish further evidence of a heterogeneity of isolated native lignin. The lower

Table V. Methoxyl Content of the Lignin Fractions

Fraction	*Methoxyl, %*
Unfractionated	15.1
A	15.5
C	15.6
D	15.7
E	15.6
F	15.3
G	15.0
H	14.9
I	15.4
J	14.1

Table VI. Elementary Analyses[a] of Isolated Native Lignin Fractions

Sample	Fraction of total	Carbon, %	Hydrogen, %	Oxygen[b], %
Unfractionated	....	65.30	5.88	28.82
II-2	0.41– 1.70	64.44	5.32	30.24
II-3	1.70– 2.98	64.11	5.87	30.02
II-5	4.07– 5.61	64.89	5.83	29.28
II-8	10.08–12.0	64.88	5.49	29.37
II-11	16.2 –18.5	65.58	5.68	28.74
II-14	22.31–24.3	66.61	5.67	27.72
II-17	28.8 –30.4	67.27	5.60	27.13
II-20	33.9 –36.8	66.81	5.66	27.53
J	21.1	61.78	5.49	32.73

[a] Oven-dry basis.
[b] Calculated by difference.

molecular weight fractions tend to have a lower methoxyl and a different carbon and hydrogen content as compared with the higher molecular weight fractions.

ABSORPTION SPECTRA

To gain more complete information about the characteristics of the fractions, their absorption spectra were determined. The different absorption spectra—ultraviolet, visible, and infrared—offer a very good means of determining whether or not the fractions are similar. As Jones (*9*) points out: "A given organic compound, if exposed to continuous radiation, might show strong absorption in the ultraviolet region associated with shifts in electronic energy levels in the structure; might appear strongly colored in the narrow band of visible radiation because of resonant absorption within so-called chromophoric groups and it certainly would show a characteristic absorption pattern in the infrared region as a result of resonant vibration of atomic and molecular groups." Each compound or mixture of materials has a specific and unique absorption which is in effect a "fingerprint" of that particular material (*22*).

Visible Spectra

The visible spectra of the various fractions provided a quantitative description of the color of the fractions in solutions of dioxane. The absorption was determined in the spectral region of 400 to 700 mmu using the Beckman quartz spectrophotometer, Model DU. For both the visible and the ultraviolet regions, the data were recorded in terms of the specific extinction of the solution calculated from the equation $k = E/cd$, where E was the extinction or $\log I_c/I$, I_c the incident intensity, I the transmitted intensity, c the concentration in grams per liter, and d the thickness of the cell in centimeters. Specific extinction, therefore, is the extinction of a substance for unit concentration and thickness. These data for the visible region are tabulated in Table VII.

The visible absorption spectra of the fractions were continuous and not characterized by the presence of any bands. The absorption decreased from 400 to 700 mmu, the general relationship between the wavelength and the specific extinction for each fraction being similar. As the molecular weight of the fraction decreased, the specific extinction of the fractions tended to decrease also. In other words, the solutions of the fractions in dioxane were more deeply colored for the higher molecular weight constituents than for the lower molecular weight constituents.

Table VIII. Comparison of the Ultraviolet Absorption Spectra for Isolated Native Black Spruce Lignin and Its Fractions

	mmu				
Sample	325	280	260	235	282
	ek, l./g. cm.				ϵ, l./mole × cm. × 10^{-4}
Unfractionated	5.0	18.8	14.2	47.5	7.9
A-6	4.6	21.7	17.0	43.8	14.6
C	4.3	16.7	8.6	32.5	7.8
E	5.1	17.5	9.3	31.1	8.0
F	4.3	18.2	14.3	41.5	8.8
G	4.7	18.3	13.9	43.5	7.5
I	5.6	18.3	14.5	42.5	5.1
J	5.0	17.3	15.8	39.5	5.9

In connection with his studies concerning the effect of the bleaching of eastern spruce groundwood with sodium peroxide, G. Jones (*18*) compared the visible spectra of dioxane solutions of unbleached and bleached isolated native lignin. The relationship which he found between the bleached and the unbleached lignin was similar to the relationship found between the high and the low molecular weight materials—i.e., the unbleached lignin had a higher specific extinction than the bleached lignin.

Ultraviolet Spectra

The ultraviolet spectra for the region from 220 to 400 mmu were also obtained, using the Beckman quartz spectrophotometer, Model DU. The specific extinction of the fractions and the unfractionated lignin at selected wavelengths are tabulated in Table VIII. From these values no marked trend was evident. Fraction A-6 had a higher extinction at 280 mmu. Fraction J had a relatively smaller minimum in the region of 260 mmu. Because the specific extinctions were similar without any marked trend, it was expected that the molar extinction would be directly related to the molecular weight. This was found to be the case as indicated in Table VIII.

Even though the specific extinctions of the various fractions disclosed no relation to the molecular weight, examination of the spectra led to the conclusion that the fractions were not identical. A further analysis of the ultraviolet data was made by resolving the over-all absorption envelope into its component bands as has

Table VII. Visible Absorption of Isolated Native Black Spruce Lignin

Sample	Specific extinction[a] at λ mmu						
	400	450	500	550	600	650	700
Unfractionated	0.224	0.099	0.051	0.025	0.013	0.006	0.003
A-6	0.305	0.139	0.078	0.041	0.021	0.013	0.009
C	0.260	0.116	0.063	0.032	0.016	0.011	0.008
E	0.302	0.132	0.069	0.035	0.018	0.009	0.005
F	0.277	0.110	0.055	0.029	0.015	0.008	0.006
G	0.221	0.082	0.040	0.019	0.009	0.004	0.002
I	0.222	0.074	0.033	0.013	0.005	0.004	0.004
J	0.232	0.068	0.024	0.007	0.002	0.002	0.001

[a] Units of specific extinction are liters per gram centimeters.

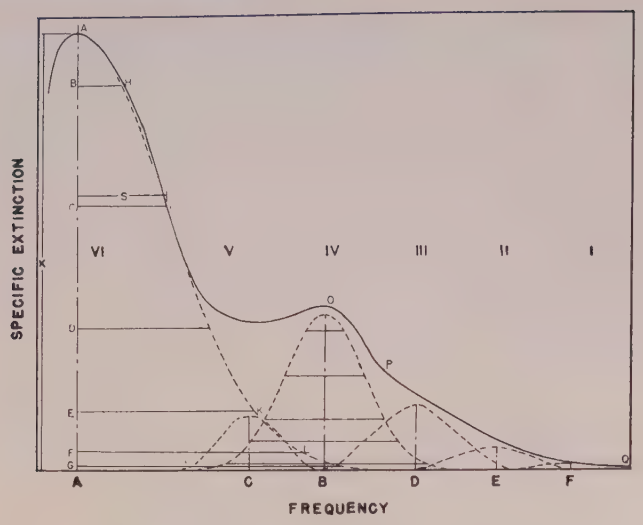

Fig. 1. Resolution of an ultraviolet spectrum into its component bands

been done for some other compounds (*23*). Trucano (*24*) has resolved the ultraviolet data from a series of lignosulphonic acid fractions into their component curves assuming a background absorption curve in the higher wavelength regions. This assumption resulted in two bands. Such an assumption did not seem to adequately fit the data at hand. Consequently, the calculation of the component curves was based upon the following assumptions which are more common to this type of calculation:

1. The component bands are symmetrical.
2. The shape of the component bands is that of a Gaussian distribution.

To make this calculation, it was first necessary to plot the absorption data on a frequency basis, either using the units of fresnels or wave numbers. A typical resolution of the ultraviolet spectrum of a fraction into its component bands is shown in Fig. 1. It was assumed that band VI appearing at frequency A was strong enough to show the component envelope from a to i. Frequency A was selected so that a Gaussian distribution fitted this portion of the curve. The half of the envelope was then drawn according to this distribution—i.e., at 0.606 k the width of the envelope is 2s, etc. The next steps were to select the position of the band IV at frequency B, to estimate the value of s using the total curve o to p as a guide, and to draw in the corresponding curve as for VI, except that the complete curve was drawn at this position. After band IV had been drawn, then the band V at C was drawn by subtracting the component bands VI and IV from the total curve. Band V was checked for symmetry and for a Gaussian shape at the 1s and 2s positions ($^1/_2$ the breadth). If the band V was not symmetrical, small changes were made in either band VI or band IV to obtain better symmetry. Usually changes were made in band IV. These alterations were to change the position of the band, to change the value of k, or to change the value of s. After a symmetrical curve had been drawn at position C, the remaining portion of the total curve from m to n was resolved into its component bands. This was done by subtracting band IV from the total curve and plotting this difference as for band V. These curves were also calculated to have a symmetrical form with an approximate Gaussian distribution.

The results of this analysis are tabulated in Table IX. The absorption spectra were resolved into six or seven bands whose positions checked quite closely from one fraction to the other. Although the analysis is of an empirical nature, it is significant that certain trends were observed as a result of the analysis. Two absorption bands, one at 38,300 cm.$^{-1}$ (261 mmu) and the other at 32,120 cm.$^{-1}$ (311 mmu), showed a tendency toward high specific extinction values as the molecular weight of the fractions decreased. The ultraviolet absorption spectra of the fractions indicated that the structural configurations contributing to the ultraviolet absorption spectra were repeated among the fractions. The repeating units leading to higher molecular weights did not appear to be identical, but the basic chromophoric structure might be similar as indicated by the relatively constant band positions observed in the over-all absorption spectra. Differences in substitution on this basic structure might contribute to the differences observed among the fractions.

A possible harmonic relationship between the bands disclosed by the above analysis was suggested, as well as a possible relationship to some fundamental frequency in the infrared region. Glading (*17*) suggested that the carbonyl group in the native lignin building unit could be responsible for the band at 352 mmu in the absorption spectrum of the phenylhydrazone of isolated native black spruce lignin. An almost complete enolization of the carbonyl group could account then for the absence of this band in the spectrum of isolated native lignin. Jones (*8*) observed that when isolated native spruce lignin was methylated with methyl alcohol-hydrochloric acid, the band at 1663 cm.$^{-1}$ in the infrared spectrum was eliminated; therefore, he assigned this band to a ketone or an aldehyde carbonyl group. If Glading's hypothesis is correct, then the component bands for a methanol-isolated native lignin should be appreciably different from those for the isolated native lignin—i.e., the absorption in this region should decrease. The ultraviolet spectrum of the same preparation of methanol-isolated native lignin as used by Jones was obtained and analyzed into its component bands. When compared with the spectrum of isolated native lignin, it was found that the methylation caused a small increase in the absorption from 400 mmu to about 260 mmu. The component bands did not show any significant correlation. It was concluded that these bands in the 300 to 400 mmu region of the ultraviolet absorption spectra were not directly related to the carbonyl group as is the band at 1663 cm.$^{-1}$. The component bands in the ultraviolet spectra may be related to other bands in the infrared spectra; however, it was not possible to make any correlation with known structures and known spectral assignments.

Infrared Spectra

A Perkin-Elmer Model 12-B infrared spectrometer fitted with a General Motors breaker-type amplifier and a Brown strip chart recorder was used to obtain the infrared absorption spectra. A sodium chloride prism was employed to cover the range from 2.5 to 14 mu.

Table IX. Analysis of the Ultraviolet Spectra of Fractions of Isolated Native Black Spruce Lignin

Sample	Band VI		Band V		Band IV		Band III		Band II		Band I	
	ν[a]	K	ν	K	ν	K	ν	K	ν	K	ν	K
Unfractionated	1332	58.5	1140	5.7	1060	18.4	970	4.7	925	4.7	880	3.0
A-6	1320	47.2	1144	2.5	1065	21.0	952	4.5	893	3.2	837	1.5
C	1292	34.5	1150	3.2	1065	16.7	962	4.5	912	3.5	862	1.7
E	1287	31.2	1156	2.5	1062	17.5	957	4.7	900	4.1	844	1.7
F	1300	42.4	1150	5.2	1065	18.0	965	4.4	918	4.5	867	2.2
G	1327	50.5	1144	6.0	1068	17.5	962	6.0	..	..	858	1.5
I	1332	49.2	1143	2.8	1062	17.5	958	6.0	893	4.2	832	1.4
J	1332	46.0	1150	5.8	1070	16.4	975	7.0	893	2.5	..	..
Av.	1315 ± 17		1147 ± 9		1065 ± 5		963 ± 12		905 ± 20		854 ± 22	
cm.$^{-1}$	43,833		38,233		35,500		32,100		30,166		28,466	
mmu	228		262		282		312		331		351	

[a] ν = fresnels.

The procedure developed by Jones was used (*8*). The films were prepared "by filling a shallow cup comprised of a metal ring and salt plate base with absolute ethyl alcohol, adding a small proportion of dioxane solution, and allowing evaporation to take place slowly." Most of the films prepared were strongly adherent clear films, nearly transparent to visible light. In some a cloudiness developed in the film after it was prepared; however, this did not seem to effect the resolution of the infrared bands.

The transmittance was calculated manually as the ratio of the energy of the recorded sample to the background energy curve. The light-box computer of Willis and Philpotts (*26*) as modified by Spearin (*25*) made the computation quite rapid and reasonably accurate.

As has already been discussed, the infrared absorption spectra is more indicative of the chemical linkages which are present in the molecule than either the visible or the ultraviolet absorption spectra. Comparison of the infrared absorption spectra of the fractions shown in Fig. 2 indicated that the fractions contained essentially the same structural elements. There were no marked changes to be observed in the bands present in the unfractionated as compared with the fractionated lignin. Although there appear to be no new bands introduced into the lignin spectrum or eliminated therefrom, a change in the bands occurring in the carbonyl region was observed. In the unfractionated material carbonyl bands were present at 1760 and 1664 cm.$^{-1}$. The high molecular weight fractions showed only a broad inflection in the region from 1700 to 1760 cm.$^{-1}$ together with the band at 1665 ± 5 cm.$^{-1}$.

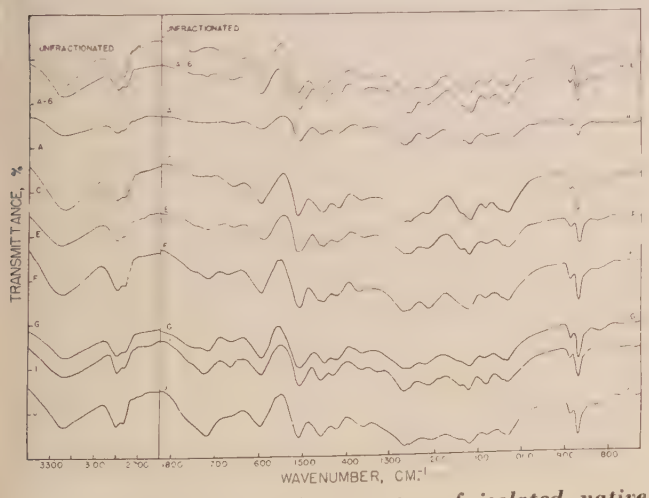

Fig. 2. Infrared absorption spectra of isolated native black spruce lignin fractions

50% transmittance level is indicated for each curve.

As the molecular weight of the fractions decreased, a band developed at 1710 to 1722 cm.$^{-1}$, an inflection remained at 1750 ± 5 cm.$^{-1}$, and, finally, the band at 1665 cm.$^{-1}$ was reduced in magnitude until it was only an inflection. This trend held without exception as the molecular weight decreased. Because the data have only qualitative value, it was difficult to say with certainty what the relationship between the fractions was with respect to the specific extinctions. Comparison of the curves indicated that the high molecular weight materials may have had a lower extinction than the lower molecular weight materials in the carbonyl region.

It is interesting to note the hypothesis developed by G. Jones (*18*) in which he stated ". . . lignin is composed of two or more structurally dissimilar fractions. It is suggested that the structural difference between these fractions may lie, at least in part, in the positions of the carbonyl groups (and possibly the phenolic hydroxyl groups) relative to the rest of the lignin structure. Since the carbonyl group is commonly considered to be a chromophore in many organic compounds, it is conceivable that the above situation could cause a variation in color among the several lignin fractions." The results of this work point toward a verification of this hypothesis—i.e., isolated native lignin is composed of several dissimilar fractions differing in their molecular size, in their color, and their chemical structure. The infrared absorption data indicate that the structural differences among the fractions occurred primarily in the carbonyl structure.

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The Role of the Hemicelluloses in the Mechanism of Wet Strength

MANUEL N. FINEMAN

Data are presented which confirm earlier concepts that the alkali-soluble, water-swellable, hemicelluloses are responsible for interfiber bonding and, hence, are a major factor in the strength of paper. The major factor in the mechanism of wet strength is shown to be the insolubilization and reinforcement of these hemicellulosic constituents by the wet-strength additives. The mechanisms of tear strength and of softness in paper are also considered in terms of the role played by the hemicelluloses, and conditions which may promote improvements in these properties are suggested.

The mechanism of wet strength in paper has long intrigued investigators and has led to much speculation and an abundance of publications. These publications have been abstracted by Collins and Adrian in their recent excellent review of the literature on wet strength (*7*) and reveal the wide divergence of opinion which exists. However, since wet strength may be produced by different materials, in different ways, it is not surprising that no one general theory seems to fit all cases.

The purpose of this report is to call attention to some observations into the role of the hemicelluloses in paper. It is hoped that these observations will promote a crystallization of existing ideas as to the mechanism of strength in paper, and also suggest a general concept for the mechanism of wet strength.

THE HEMICELLULOSES

It has been known for some time that the hemicelluloses have a marked influence on pulp quality and pulp properties (*15*, *35*). Their presence has been correlated with swelling (*11*, *28*, *33*) and they have been shown to influence the bonding of fibers (*29*). March (*22*) effected a stepwise removal of alkali-extractable hemicellulose from holocellulose and was able to correlate strength properties with hemicellulose content. The effect of the hemicelluloses can also be noted in the burst (*12*), tensile (*21*), tear (*3*), fold, opacity, and specific surface of the paper sheet (*34*).

Until recently much of the work on hemicelluloses in this country has been connected with studies of their chemical composition (*35*) whereas investigators such as Jayme (*18*, *19*, *20*), Markila (*23*), Greenane (*12*), and others of Europe have devoted attention to their effect on the physical properties of paper.

The hemicelluloses which are considered in this work may be defined from the physical-chemical point of view as those cellulosic portions of pulp that are soluble in alkali and hydrolyzable by dilute acid. From the organic-chemical point of view this alkali-extractable fraction includes cellulosic constituents of a wide range of degree of polymerization — up to about 200 D. P. (*26*), and comprises such materials as polymeric pentoses and hexoses, polyuronides, polyuronic acids, sugar residues, short cellulosic chains, etc. The hemicelluloses are swellable in water and, in some cases, are water soluble. They constitute up to 15% by weight of sulphite and kraft stocks and about 2 to 3% of rag stocks.

PAPER STRENGTH

There are essentially two very different cellulosic phases in pulp: a highly crystalline phase which constitutes the bulk of the cellulose fiber, and a relatively amorphous, water-sensitive, hemicellulose phase. The hemicelluloses swell readily in water and render the fiber surfaces tacky so that, upon drying, they are strongly adhered (*10*). Adhesion may be due to intercrystallization of the two cellulosic phases, or merely an amorphous type of fusion. Thus the hemicelluloses may be regarded as the interfiber adhesive in paper. Moreover, the hemicelluloses are presumed to reswell whenever exposed to water thus accounting for the poor wet strength of paper.

The important characteristic here is not that the hemicelluloses are present, *per se*, but rather that they swell in the medium used for preparation of the paper (*10*). Paper prepared in a medium in which the hemicelluloses do not swell will lack strength unless a material is present which can swell in that medium and thus act as the interfiber adhesive.

Beaten sulphite and rag pulps were treated with cold 17.5% sodium hydroxide to remove the hemicellulosic fraction. The extracted pulps were then washed and made into handsheets; and their strength properties compared with those of sheets made from the untreated pulps.

Table I shows that paper made from extracted pulp

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Table I. Effect of Alkali Extraction of Pulp on Handsheet Properties

Pulp	Sheet weight, g.	Tensile, 2-in. span Dry	Tensile, 2-in. span Wet	Tensile, zero span Dry	Tensile, zero span Wet
Sulphite	2.64	6.5	0.64	9.9	6.8
Extracted sulphite	3.31	1.1	0.20	7.3	7.5
Rag	3.19	11.4	0.86	12.1	10.7
Extracted rag	3.51	3.0	0.25	9.0	7.7

had very low wet and dry tensiles (2-in. span, 1-in. strips). In fact removal of the hemicelluloses resulted in a decrease in dry strength to about one-fifth that of the original. Moreover zero span values indicate that this decrease was not due to fiber degradation since the fibers themselves still retained much of their original strength (Fig. 1).

To check this latter point further, Table II shows the effect of addition of cooked starch to the papers via the tub. Only 3% starch more than restored the dry tensile to the rag paper and helped the sulphite considerably. Since the alkali-extractables were about 2% of the rag pulp and about 14% of the sulphite, the difference in their response to the starch may be rationalized (Fig. 2).

As noted earlier, it is commonly recognized that the hemicelluloses are an important factor in the strength of paper; and the above data offer a striking corroboration of this general concept.

RESIN TREATMENT

The response of these papers to wet-strength resin treatment was examined. Resins were applied to the sheets made from the original and extracted pulps as well as to the extracted rag pulp to which starch had been added. Resin application was by tub-size addition in order to avoid complications due to possible changes in retention which might be encountered in beater additions. The resin-treated sheets were fully cured at 150°C. for 15 minutes.

The results, reported in Table III, show that, in the absence of the hemicelluloses, the wet-strength resins were totally ineffective. Although results based upon calculations of per cent wet strength may appear to be high, they are quite misleading in view of the unusually low, and variable, dry strength values. The actual wet strength of papers made from extracted pulps was so low that it must be concluded they did not respond to the wet-strength treatment (Fig. 3).

These results show that the major factor in the mechanism of wet strength is the interaction of the resins with the pre-existing interfiber bonds in the paper rather than the establishment of new resin bonds between fibers.

In rag stocks, where the fibers are exceptionally long and the degree of intertwining of fibers is great, a very small amount (2%) of interfiber adhesive was sufficient to bond the fibers. Similarly, addition of resin to fractionated rag pulp permitted establishment of a small number of resin bonds between the fibers—sufficient to show a slight improvement in paper strength (Fig. 4).

Table II. Effect of Starch on Properties of Handsheets

Pulp	Sheet treatment	Pickup, %	Tensile, 2-in. span Dry	Tensile, 2-in. span Wet
Sulphite	3% starch	2.86	13.2	0.52
Extracted sulphite	3% starch	..	4.6	..
Rag	3% starch	2.81	21.4	1.02
Extracted rag	3% starch	3.94	14.5	0.55

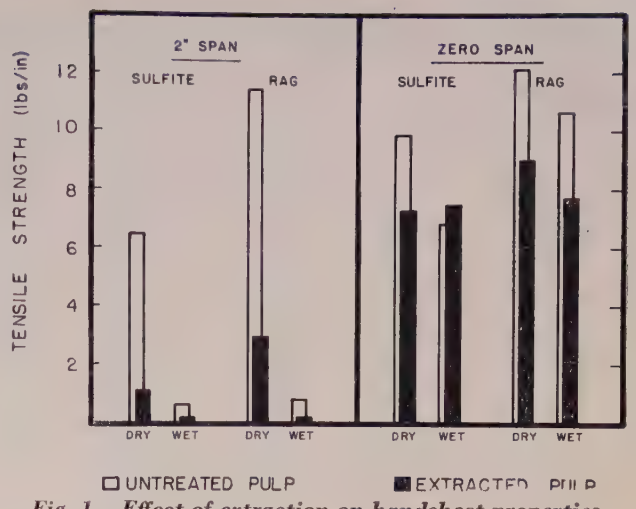

Fig. 1. Effect of extraction on handsheet properties

Replacement of the hemicelluloses by cooked starch produced paper of high dry strength which responded well to wet-strength treatment. This confirms the similarity in behavior between the hemicelluloses and other polysaccharide adhesives noted previously.

A few experiments involving the addition of wet-strength resins to the fractionated pulps via the beater are reported in Table IV and confirm the results shown in Table III. Further they indicate that retention of resin in the beater may also be somewhat dependent upon the presence of the alkali-soluble component of the pulp.

In the light of the above data it is now appropriate to re-examine the various phases of papermaking and determine the manner in which the hemicelluloses play such a significant part.

When pulp is beaten the fibers are cut and abraded causing the formation of many fibrils on the fiber surface. At the same time the pulp imbibes water and swells. The net result is an observed increase in the specific surface of the pulp and also in its wetness. It has been noted that the hemicelluloses play a principal role in this beating process, although the exact nature of that role has not been altogether clear (*6*, *24*). As indicated earlier, their presence has been correlated with swelling and they are known to shorten beating time. For example, a rapid increase in the wetness of a pulp may be obtained by adding pentosans, or guar (*30*) to the beater. Moreover rag pulps, which contain only a very small amount of hemicellulose, are very difficult to beat.

The data reported here add weight to the belief that beating is desirable in preparation of pulp only because it is a simple mechanical means for producing an increase in swelling. Mechanical working of the fiber involves a loosening of the whole fiber structure accompanied by wetting of the cellulosic phases. The highly crystalline cellulose, lacking in free valences, is relatively little affected by the action of water. However, the amorphous hemicelluloses rapidly imbibe water and swell. Prolonged beating produces a pulp of a relatively high degree of wetness and results in the formation of a very dense transparent sheet of high strength such as glassine. However, prolonged beating

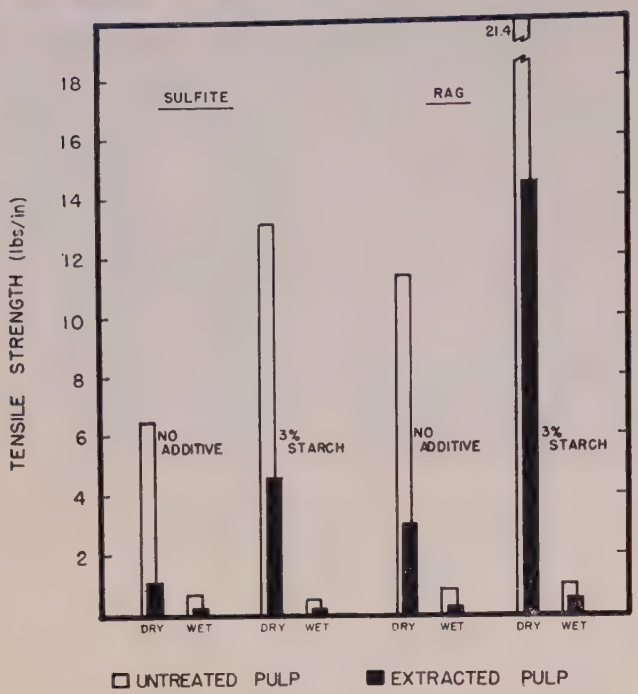

Fig. 2. Effect of starch on handsheet properties

also cuts the fibers causing a decrease in fiber length, and in paper strength. Therefore, for optimum strength, time of beating must be adjusted to achieve maximum wetness with minimum fiber damage.

Similarly, because of variations in wetness of the stock, time of beating will also affect opacity, bulk, absorbency, finish, etc., of the final product.

PAPER FORMATION

When pulp fibers are formed into a mat on a paper machine, and pressed, the swollen hemicelluloses pervade the entire structure of the wet web and are pressed into contact with neighboring fibers. As the web is dried, these hemicelluloses gradually lose their imbibed water and shrink, drawing adjacent fibers and fibrils, with which they are in contact, close together (*5*). Thus, in the dry state the hemicelluloses act essentially as fiber adhesives and, by intercrystallization, or by fusion, form tough cellulosic bonds between fibers. These investigations confirm that, under normal conditions, no fiber bonding is possible in the absence of these hemicelluloses. Under abnormal conditions, however, such as application of extreme pressures to swollen fibers during the drying cycle, some interfiber bonding may result (*8*).

In rag pulps, where the fibers are exceptionally long and the degree of fiber intertwining is very great, a small amount of hemicellulose is sufficient for interfiber bonding and strength development. Thus only 2% hemicellulose in rag permits formation of some of the strongest papers known. Removal of this small amount, however, eliminates interfiber bonding and results in a marked decrease in paper strength. Moreover substitution of the 2% hemicellulose by 4% swollen starch, a material very similar to hemicellulose both physically and chemically, results in a net increase in dry strength to almost double the normal value.

Thus it is concluded that the dry strength of paper is a function of the length and strength of the pulp fibers, as well as the amount of hemicellulosic or other interfiber bonding material which is present.

In view of the above, improvements in dry strength may be readily obtained by increasing the number of long and strong fibers, by increasing the number of interfiber bonds, or by reinforcing existing bonds so as to increase their strength. Upgrading of pulp for strength improvement is a common practice. Addition of fiber adhesives such as cooked starch or various thermoplastic additives improves dry strength without necessarily affecting wet strength. Among others, cellulose ethers (*9*), guar (*30*), glue, and various protein gums have already been used for this purpose. Strength improvement by reinforcement of existing bonds will be discussed below.

WET STRENGTH

Although the hemicelluloses are responsible for dry strength in paper, they are readily reswellable in water. Consequently they are also responsible for the lack of wet strength in paper. Therefore it is suggested that the only requirement for wet strength is that the hemicelluloses be made nonwater-swellable or, at the very least, less water-swellable than usual. Further, any material which will interact, either physically or chemically, with the hemicelluloses so as to reduce their degree of swelling in water, will be suitable for use as a wet-strength additive.

In recent years Bursztyn (*4*), Steenberg (*16*, *17*, *33*)

Table III. Response of Sheets to Wet-Strength Resin Treatment

Handsheets	*Resin*	*Pickup, %*	*Catalyst*	*pH*	*Tensile, 2-in. span* *Dry*	*Wet*
Sulphite	Uformite 467	4.79	Alum	4.0	14.5	7.34
Extracted sulphite	Uformite 467	..[a]	Alum	4.0	2.2	0.81
Rag	Uformite 467	4.48	Alum	4.0	16.6	11.68
Extracted rag	Uformite 467	..	Alum	4.0	5.8	3.01
Sulphite	Uformite 470	4.47	HCl	4.0	11.3	5.26
Extracted sulphite	Uformite 470	..	HCl	4.0	2.0	1.11
Rag	Uformite 470	3.91	HCl	4.0	19.5	14.41
Extracted rag	Uformite 470	..	HCl	4.0	3.1	2.63
Sulphite	Uformite 475	4.84	HCl	4.0	9.8	5.14
Extracted sulphite	Uformite 475	..	HCl	4.0	0.7	0.28
Rag	Uformite 475	4.37	HCl	4.0	18.1	7.53
Extracted rag	Uformite 475	..	HCl	4.0	3.2	1.07
Sulphite	Amberlite W-1	4.36	..	...	18.5	9.22
Extracted sulphite	Amberlite W-1	..	..	...	...[b]	...
Rag	Amberlite W-1	4.03	..	...	27.0	16.35
Extracted rag	Amberlite W-1	5.79	..	...	2.9	2.07
Rag + starch	Uformite 467	3.58	Alum	4.0	18.1	11.70
Extracted rag + starch	Uformite 467	5.13	Alum	4.0	13.2	5.99

[a] Sheets too weak to be left unsupported for weighings. Pickup ca. 5%.
[b] Sheets fell apart.

nd Salley and Blockman (*31*) have indicated that wet-trength resins operate by forming water-resistant esin bonds between fibers. However, the data re-orted here show that resins cannot strengthen pulps rom which the hemicelluloses had been removed. onsequently it must be concluded that the resins perate principally by interaction with the hemicel-uloses, and do not themselves form interfiber bonds.

Wet-strength resins not only impart wet strength to paper but also cause appreciable improvements in dry trength. Since they do not form interfiber bonds, he role of resins in improving dry strength must be hat of reinforcing the hemicellulose phase, i.e., making he natural interfiber bonds in paper stronger or tougher. onsequently, it is suggested that the dry strength, s well as the wet strength, of paper may be improved y judicious selection of materials which reinforce and nsolubilize the cellulosic interfiber bonds.

Most of the materials currently used as wet-strength additives in paper are thermosetting resins such as the condensates of urea, melamine, or phenol with formal-dehyde. They are applied in the partially condensed state and are converted by heat to the insoluble and infusible fully condensed state. In the conversion process these resins interact with the hemicelluloses, either by chemical reaction, or by mere physical mixing, or both, reinforcing them and making them resistant to the swelling action of water. Physical interaction can make the interfiber bonds stronger, much as concrete may be reinforced by chicken wire, and thus cause improved dry strength. Physical interaction, with or without chemical interaction, can reduce the tendency of the hemicelluloses to swell in water and thus can improve wet strength.

There is reason to believe that a significant part of the interaction of resins with hemicellulose is chemical. For example, formaldehyde (*14*) and glyoxal (*1*), both monomers, and both active cellulose reactants, have been used commercially to produce wet strength in paper. Papers which have been subjected to severe heat treatment, and pulps which have been oxidized (*32*), develop some wet strength even without synthetic additives. Consequently it is suggested that any material, monomeric or polymeric, which will react with the hemicelluloses so as to decrease their tendency to imbibe water and swell, will impart wet strength to paper. For nonthermosetting materials heat treatments to cure the additive should be more accurately considered as heat of activation for reaction of the additive with the hemicellulose.

In the waterproofing of starch by resin treatment, it is generally accepted that an actual chemical interaction exists—probably between the methylol groups of the resin and the hydroxyl groups of the polysaccharide (*2*). A similar interaction of resin with hemicellulose is very probable.

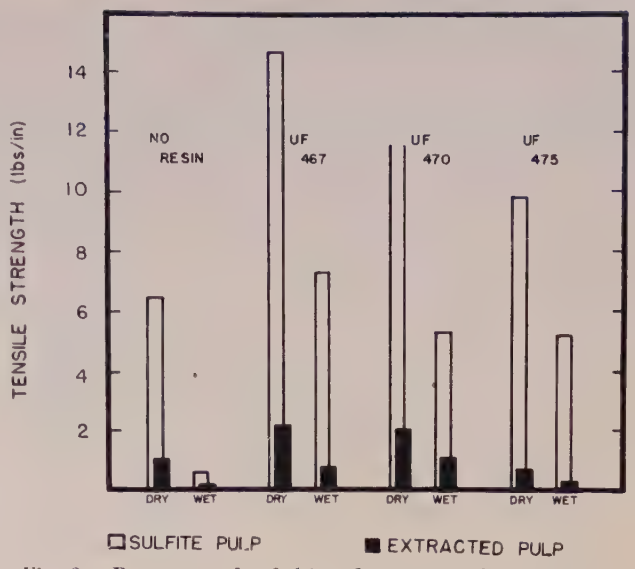

Fig. 3. Response of sulphite sheets to resin treatment

Moreover, it has been reported that certain irreversible changes occur in hemicellulose on drying which cause the known lowered strength of paper made from dried pulps. These changes also affect response of pulp to wet-strength treatment. Unpublished data, obtained by the writer, have shown that wet-strength resins are much more effective on slush pulps than on similar pulps prepared from dried laps. Apparently the hemicelluloses in dried pulps have been altered, either chemically or physically, so that even after rewetting the extent of their interaction with additives is reduced.

However, chemical interaction of hemicellulose with monomeric, i.e., nonthermosetting additives will not necessarily improve dry strength nor permit development of the maximum wet strength attainable with a thermosetting resin. For example, formaldehyde improves wet tensile sixfold but does not alter dry tensile (*13*). Thermosetting resins improve wet tensile tenfold; and dry tensile, by a factor of 1.5 to 2. For development of maximum strength, thermosetting resins are the preferred additives rather than monomeric reactants. During their condensation these resins probably interpolymerize with the available hydroxyl groupings of the hemicelluloses and produce a mixed polymer of resin and cellulose. In such cases physical as well as chemical interaction will result and will not only reinforce the interfiber bonds and improve dry strength but will also make these bonds less water-swellable, thus improving wet strength as well.

Finally it should be emphasized that this interaction is impossible in the absence of hemicellulose. As noted earlier, the hydroxyl or other groupings in crystalline cellulose are strongly bound into the crystal

Table IV. Effect of Beater-Added Resins on Fractionated Pulps

Pulp	*Resin 3%*	*Catalyst*	*pH*	*Sheet weight, g.*	*Tensile, 2 in. span*[a] *Dry*	*Wet*	*N, %*
Sulphite	Uformite 467	Alum	4.0	2.30	9.2	2.29	...
Extracted sulphite	Uformite 467	Alum	4.0	2.32	1.6	0.32	...
Sulphite	Uformite 470	HCl	4.0	2.39	9.1	2.33	0.35
Extracted sulphite	Uformite 470	HCl	4.0	2.23	2.6	0.59	0.18
Sulphite	Uformite 475	HCl	4.0	2.49	9.3	2.31	0.14
Extracted sulphite	Uformite 475	HCl	4.0	1.92	1.9	0.46	0.07

[a] After 28 days' natural aging.

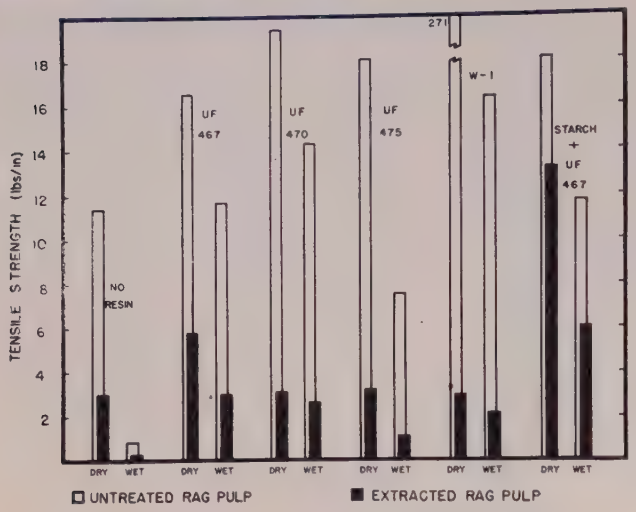

Fig. 4. Response of rag sheets to resin treatment

lattice and lack free valencies or the capacity to interact with additives.

It is now of interest to examine the effect on other properties of paper, such as tear strength and softness.

TEAR STRENGTH AND SOFTNESS

Tear strength in paper, as in fabric (*13*), depends upon the strength and extensibility of the fibers and interfiber bonds. It is at a maximum when the fibers have high intrinsic strength and also a fair degree of mobility relative to each other.

Papers such as sulphite and kraft will have relatively low tear strength because the short fibers are bound together by a large number of rigid, nonextensible hemicellulosic bonds. However, rag papers which more nearly approximate the multifilamentous character of fabric, will have good tear strength because the small number of hemicellulosic linkages permit the long fibers to have some mobility relative to each other. In view of the above, any improvement in dry strength which involves the creation of new interfiber bonds, or the addition of resin to reinforce existing bonds, will contribute to the rigidity of the paper and cause a corresponding decrease in tear strength. Assuming that fiber strength is fixed, then tear strength in paper may be improved by substitution of elastic interfiber bonds for rigid hemicellulosic linkages, i.e., by use of rubber latices or some other elastic binder (*27*), or, by reduction in the number of rigid linkages.

The hemicellulosic bonds in paper will also influence the softness of the sheet. These linkages are tough and rigid and impart stiffness as well as strength to the paper. For example, sheets made from lightly beaten pulps possess few interfiber bonds and hence are soft—and weak. Prolonged beating increases bonding and produces strength—but also stiffness and rattle.

For many years glycerine and other humectants have been added to paper as softening agents. Very recently, however, McPherson (*25*) has pointed out that the hygroscopic or humectant action of such softeners has been overemphasized. Instead he has shown that the softening action is due principally to a weakening of the sheet through decreased fiber bonding. Reduction in the number of hemicellulosic linkages or reduction in the strength of these linkages, may be the principal functions of such softeners. In this work, paper made with pulp from which all the hemicelluloses had been removed was exceptionally soft—but, of course, had no strength. Consequently it is suggested that softness, as an inverse function of interfiber bonding, can only be obtained at the expense of strength. In rag pulps, however, where the amount of interfiber bonding is small and the paper approaches the multifilamentous character of fabric, softness may be achieved without too great a reduction in strength.

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Light Reflection Measurements Applicable to the Surfaces of Wallboard

R. S. HUNTER

To establish a standard method for measurement of the capacity of surfaces to reflect light, conditions of measurement must be standardized, both spectrally and geometrically. Two sets of spectral conditions are familiar to those who work with wallboard: (1) luminous conditions corresponding to the eye response, with maximum weight given to the yellow-green region of the spectrum, and (2) blue-light conditions of "paper-brightness" measurement. There are also two sets of geometric conditions widely used for reflectance measurements: (1) 45° illumination, 0° viewing, typical of the geometric conditions under which materials are examined for visual color, and (2) 0° diffuse conditions, generally used by the illuminating engineer to evaluate the efficiency of surfaces as reflectors of light. Ten or twelve different commercial instruments are used for measurements of light reflectance under various combinations of these conditions. They vary in cost from about $200 to $12,000, as well as in the variety and accuracy of measurements they make.

THE capacity of a surface to reflect light results from its microscopic structure and the refractive and absorptive properties of its ingredients. In general, this capacity varies with wavelength of light and with the directions of incidence and view involved. Its complete specification is complex, requiring as it does both spectrophotometric curves relating to color, and goniophotometric curves relating to gloss or luster.

Figure 1 shows in four spectrophotometric curves the manner in which the reflectances of white, yellow, gray, and brown surfaces vary with wavelength throughout the visible spectrum. Note particularly that yellow and brown are characterized by low reflectance at the blue end of the spectrum.

Figure 2 is taken from an earlier paper (*6*) by the author and shows goniophotometric curves representing the reflectance in different directions of four white surfaces. These differences in geometric distribution of reflected light correspond to differences in gloss or luster.

From these spectrophotometric and goniophotometric curves, it can be seen that the reflectance of a single surface is a many-valued function varying with both wavelength and directions of illumination and view. If the reflectance of this surface is to be represented by a single number, standard spectral and geometric conditions of measurement must be adopted for its measurement.

Spectrally, two different types of functions have been applied to materials in the paper and construction fields: (1) The luminosity function covering the whole spectrum with maximum response in the yellow-green region. This function represents the relative visual weights given by the human eye to different wavelengths. (2) The "paper-brightness" type of function restricted to the blue end of the spectrum.

The luminosity function is properly used wherever matters of visual lightness or conservation of light are involved. The "brightness" type of reflectance measurement in the blue end of the spectrum is properly used wherever one is concerned with the presence of lignin or other yellowish impurities which absorb blue light and thus prevent the production of an end product of good whiteness even though blue dye is used to counteract the yellowness.

As is shown in Fig. 3, there are actually two spectral functions of each type. The luminosity curve differs with the illuminant to which it applies. Thus, in Fig. 3, are shown (Y_C) the luminosity curve for ICI standard illuminant C representing daylight, and (Y_A) the luminosity curve for ICI standard illuminant A representing incandescent lamplight. The two blue functions used for brightness measurements, are (B) the brightness function characteristic of the G.E. reflection meter and specified in TAPPI standards T 217-48 and T 452 m-48, and (Z_C) the Z or blue response function of the ICI standard observer specified in ASTM tentative method D 985–48 T.

Geometrically, three conditions of measurement are widely used in the evaluation of surface reflectance:

1. Approximate unidirectional illumination and diffuse viewing of the specimen by means of a sphere (see instruments illustrated in Figs. 5 through 9 below).
2. Approximately 45° illumination and 0° (perpendicular) viewing (see instruments illustrated in Figs. 10 through 16 below).
3. Oblique illumination and viewing in the direction of mirror reflection for measurements of gloss (not considered in the present paper).

The quantities measured by instruments of types 1 and 2 are defined as follows:

1. *Total reflectance*, R_t, is defined as that fraction of the specified incident light which is reflected by the surface. A freshly smoked surface of MgO (*10*) is the usual reference standard.
2. *45° 0° Directional reflectance*, $R_{d\,(45°\,0°)}$ is defined as the light reflected in the perpendicular direction by a surface illuminated at 45°, relative to that which would be reflected in the same manner if a perfect white were substituted for the specimen. By perfect white is meant the ideal, perfectly reflecting, perfectly diffusing, white surface which, for 45° 0° conditions, is considered to be duplicated by a freshly smoked surface of MgO (*10*).

REFLECTANCE OF WALLBOARD

For most types of wallboards it does not make much

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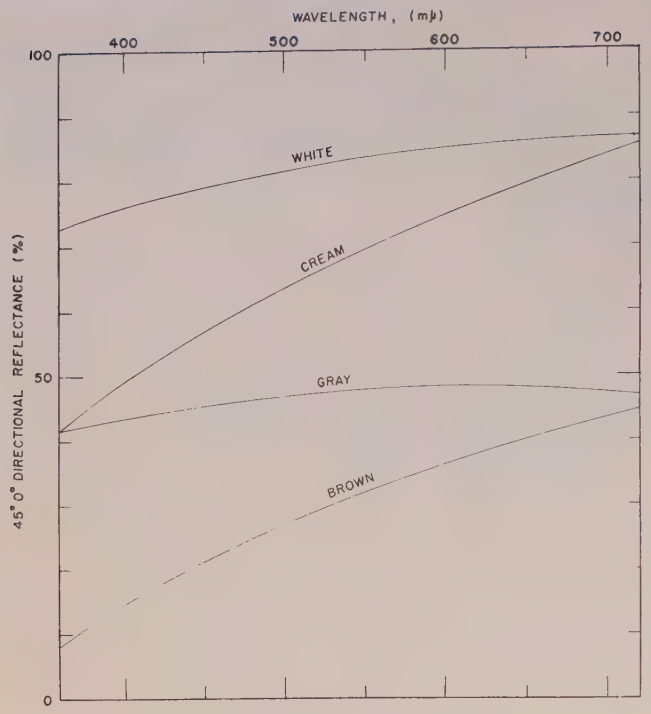

Fig. 1

difference which geometric type of reflectance measurement is used. This is because most wallboard surfaces are almost perfectly diffusing in their reflecting characteristics. For perfectly diffusing surfaces, values of R_t and R_d are the same. Spectrally, however, it does make a difference which of the above-mentioned wavelength functions is used. Wallboard is furnished in a variety of shades of white, cream, yellow, gray, and brown. Almost all vary in reflectance from one part of the spectrum to another. To show how reflectance values may change as spectral and geometric conditions of measurement are varied, the author measured a number of fiberboard specimens collected for him by J. F. Campbell. Also measured were several specimens of acoustical tile obtained by the author at the National Bureau of Standards. These were measured for 0° diffuse total reflectance with the Hazemeter (Fig. 7), an instrument intended primarily for measurements of transparency of clear plastics and glass (*1*). They were measured for 45° 0° directional reflectance with the well-known Multipurpose Reflectometer (*5*) shown in Fig. 11.

For both these instruments, there are filters corresponding to the Y_A, Y_C and Z_C functions shown in Fig. 3. We do not have a filter giving exactly the "TAPPI brightness" function but, it can be seen from Fig. 3, that results for TAPPI brightness should be but little different from those obtained with the Z_C function.

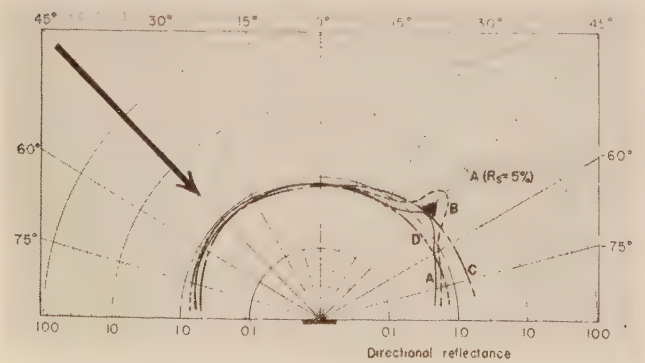

Fig. 2

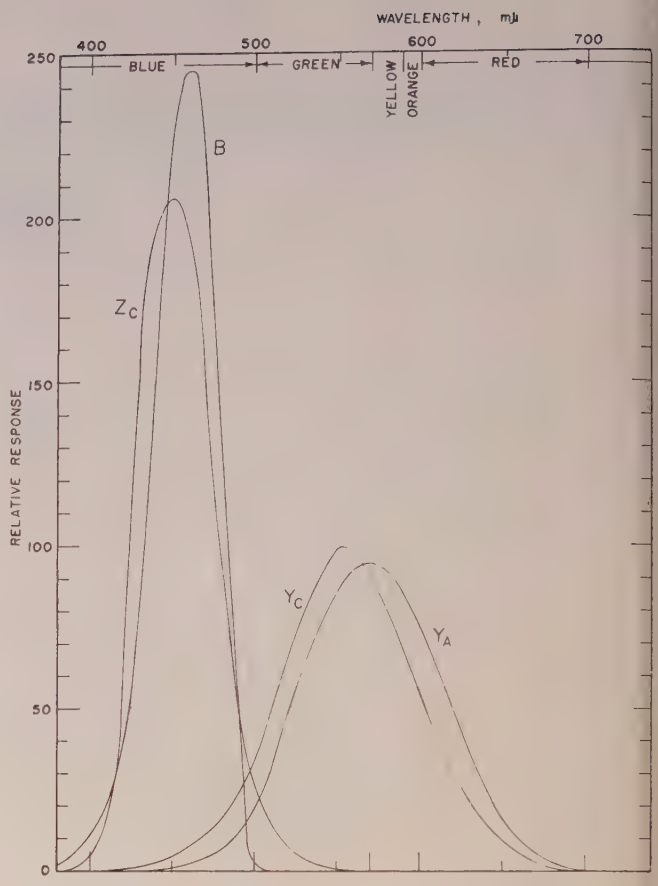

Fig. 3

The results shown in Table I are in good agreement as between the two geometries. Excepting the glossy specimen and the acoustic tile with holes large enough to interfere with reflectance measurements, there is only one instance in which corresponding values for

Fig. 4. Simple reflection meter

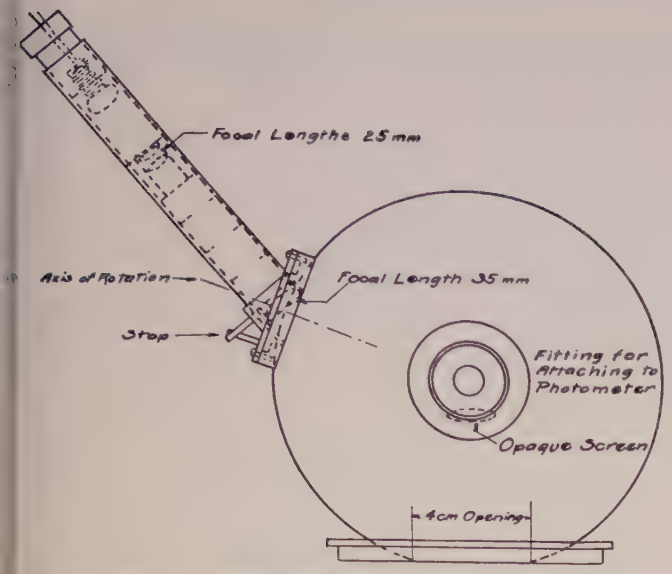

Fig. 5. Taylor 30° diffuse reflectometer

the two geometries do not agree within 1%. As noted above, agreement between the two geometries is not expected with a glossy specimen because a total reflectance value includes the effects of glossy specular reflectance and is therefore higher than the value of $R_{d\ (45°\ 0°)}$ for the same filter.

By contrast, measurements with the different filters are not in agreement with each other, particularly for those surfaces which are cream or yellowish in color. The big disparities are, of course, those between values measured according to the luminosity function with maximum weight given to the green and yellow regions of the spectrum, and those for the blue "brightness" function. There are small, but definite differences, however, between values for the yellowish

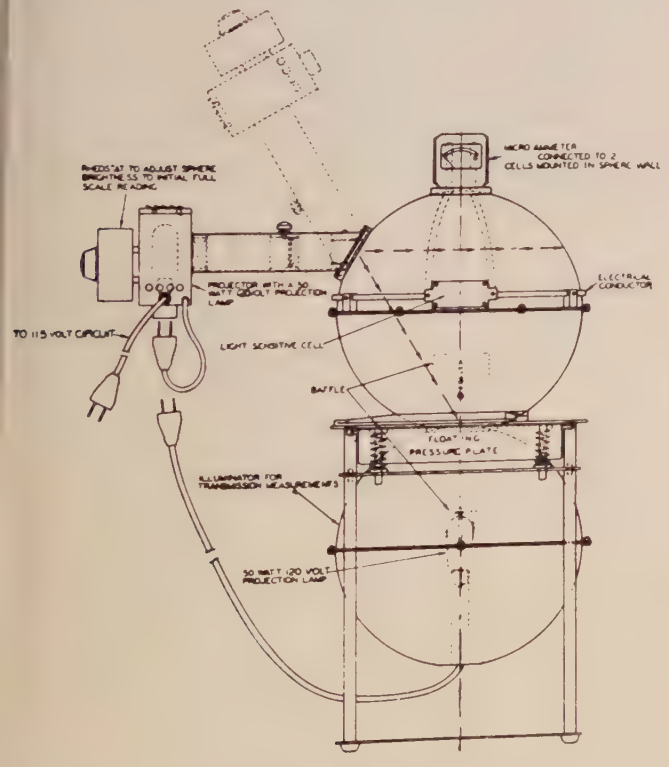
RHEOSTAT TO ADJUST SPHERE BRIGHTNESS TO INITIAL FULL SCALE READING
MICRO AMMETER
CONNECTED TO 2 CELLS MOUNTED IN SPHERE WALL
PROJECTOR WITH A 30 WATT CIRCUIT PROJECTION LAMP
ELECTRICAL CONDUCTOR
TO 115-VOLT CIRCUIT
LIGHT SENSITIVE CELL
BAFFLE
FLOATING PRESSURE PLATE
ILLUMINATOR FOR TRANSMISSION MEASUREMENTS
50 WATT 120 VOLT PROJECTION LAMP

Fig. 6. Baumgartner 30° diffuse photoelectric version of Taylor reflectometer

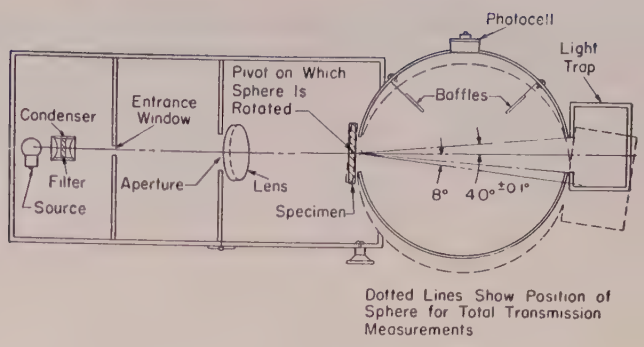

Fig. 7. Hunter pivotable-sphere hazemeter

"lamplight" and bluish "daylight" luminosity functions.

REFLECTANCE INSTRUMENTS

In Fig. 4 is shown probably the simplest reflectometer in existence. It works well on white and gray surfaces, but it takes visual skill to make lightness matches between the series of gray shades printed on this disk and slightly colored surfaces over which it is

Table I. Total (0°, Diffuse) and Directional (45° 0°) Reflectance Values of Several Wallboard Specimens for (Y_A) Lamplight Luminosity Function, (Y_C) Daylight Luminosity Function, and (Z_C) ASTM "Brightness" Function

Specimen	$R_{t\ (0°,\ diffuse)}$ Y_A	Y_C	Z_C	$R_{d\ (45°0°)}$ Y_A	Y_C	Z_C
White, painted, screen texture	80	79	72	80	79	73
White, painted, screen texture	81	80	69	80	79	69
Cream, painted, screen texture	74	71	54	74	71	55
White, painted, smooth texture	68	66	60	68	67	61
Cream, painted, smooth texture	77	74	55	77	74	56
White, painted, fibre texture	78	76	69	78	77	69
Yellow, painted, fibre texture	74	71	58	73	72	59
Yellow, unpainted, glossy texture	66	62	43	63	61	41
Acoustic tile						
White, painted, cored holes	75[a]	73[a]	68[a]	83	82	78
White, cast, porous block	69	67	62	68	68	63
Cream, cemented wood fiber	60	55	42	57	55	42

[a] Holes were large relative to test area, inclusion of one hole in each test area caused disproportionate reduction.

placed. Because of variations in amount of light reaching the test surface, results vary somewhat with direction of incident illumination. There is also a question of permanence; the writer's Reflection Gauge, given him 15 years ago, now reads almost 10% high.

A more accurate reflectance scale of this type has

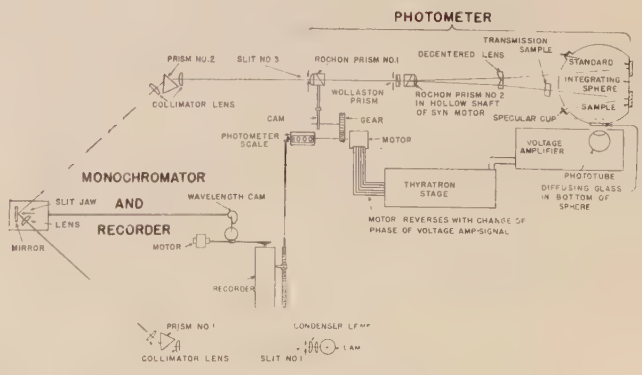

Fig. 8. General Electric Spectrophotometer

Table II. Commercial Reflectometers Measuring Total and 45° 0° Directional Reflectance for (Y_A) Lamplight Luminosity, (Y_C) Daylight Luminosity, (Z_C) ICI Blue, and (B) G.E. "Brightness." Also Rough Cost Estimate (CE) in Hundreds of Dollars

Diffuse reflectometers (sphere type)	Y_A	Y_C	Z_C	CE	*45° 0° Reflectometers*	Y_A	Y_C	Z_C	B	CE
Taylor reflectometer (*11*)	X	..	..	8	G.E. Reflection Meter (Brightness Tester) (*7*)	..	..	..	X	10
Baumgartner reflectometer (*2*)	X	..	..	4	Hunter Multipurpose Reflectometer (*5*)	X	X	X	..	6
G.E. Spectrophotometer and Integrator (*4*)	..	X	X	140	Photovolt Model 610 Reflectometer	X	X	..	..	2
Hunter Hazemeter (*1*)	X	X	X	9	Gardner Portable Reflectometer	X	X	..	..	2
PPG-IDL Color Eye	X	X	X	25	Beckman Spectrophotometer (*3*)	..	..	..	..	12
					Ansco Densitometer (*9*)	..	..	..	..	8
					Hunter Color-Difference Meter	..	X	..	..	15

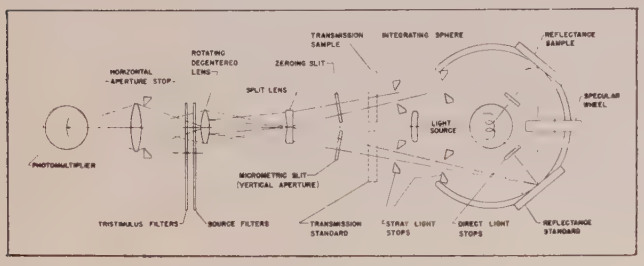

Fig. 9. PPG-IGL Color-Eye

chips of a number of colors with their values of reflectance. It is the series of 11 charts, Munsell Value Scales for Judging Reflectance (*8*), made available by the joint effort of the Illuminating Engineering Society and the Inter-Society Color Council. These charts are available from the Munsell Color Co., 10 E. Franklin St., Baltimore, Md. ($17.50).

In Table II are listed the commercially available reflectometers for R_l and $R_{d\ (45°,\ 0°)}$ most familiar to the author. In this table are X's to indicate which of the different spectral functions are easily measured by each instrument, and order-of-magnitude estimates of cost (in hundreds of dollars). It is with misgivings that the author has given estimates of cost, for most of the instruments are available in a variety of forms at a variety of prices, so that they perform a variety

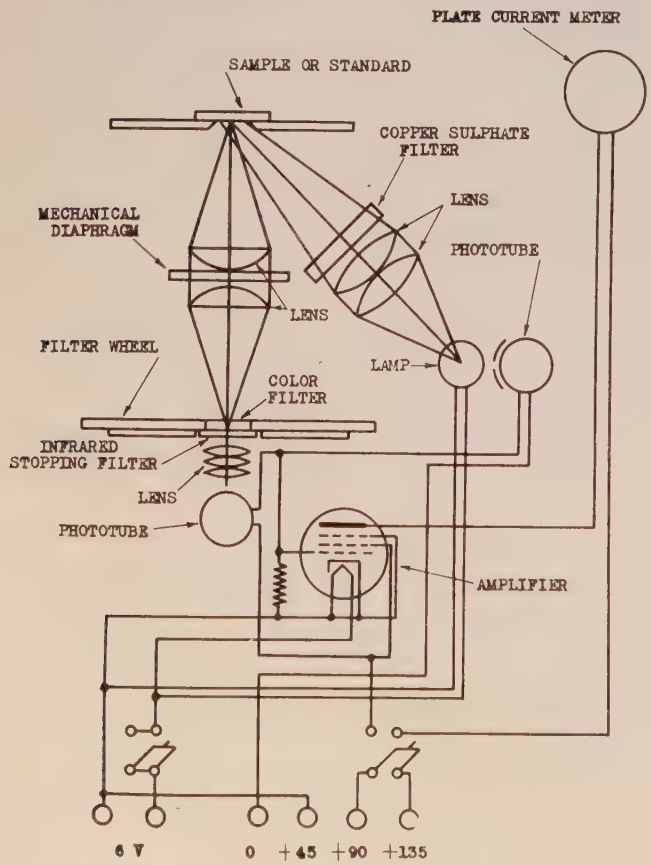

Fig. 10. General Electric 45° 0° Reflection (Brightness) Meter

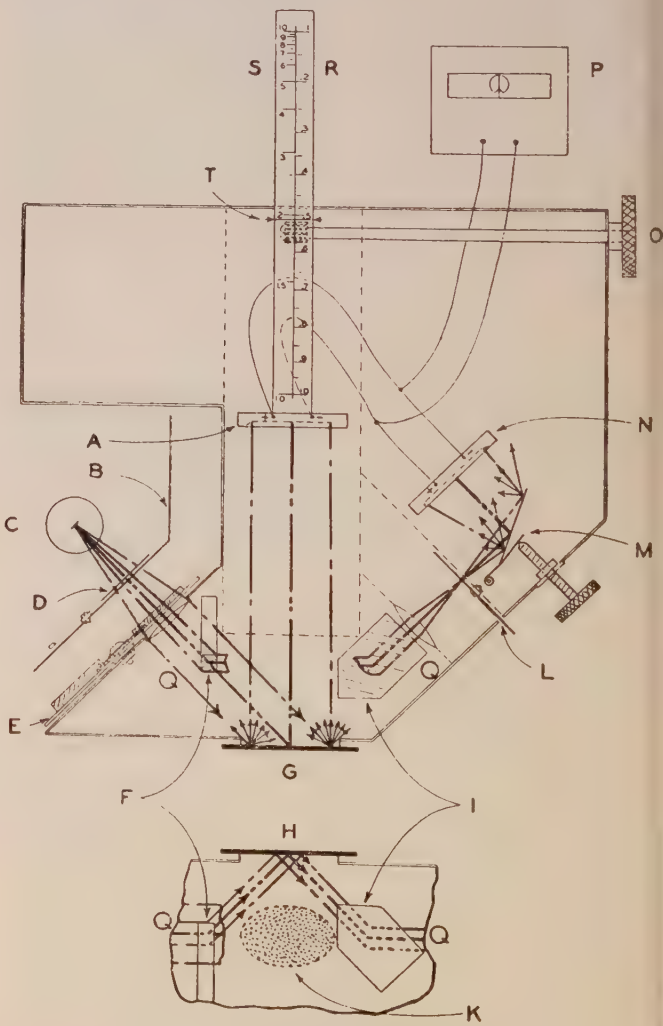

Fig. 11. Hunter Multipurpose Reflectometer (45° 0° reflectance)

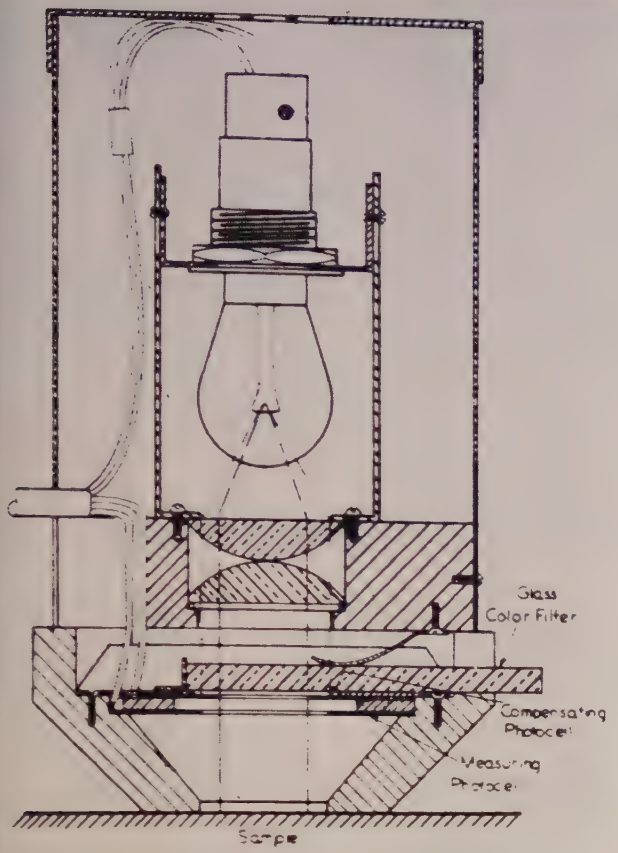

Fig. 12. Photovolt Model 610 Reflection Meter

of tasks other than those discussed here. It seems needless to point out, moreover, that these cost estimates may not include price changes made during the last few months.

Diagrams of the different instruments are shown in Figs. 5 through 16. Each of these diagrams shows the manner in which test surfaces are illuminated and viewed. Most of them also show the manners in which different spectral bands are isolated and light is measured. Noteworthy features of each instrument, and the names and addresses of their manufacturers are given in the respective legends.

Note that the direction of incidence varies between the different sphere-type instruments. It is 0° for the Hazemeter in Fig. 7, 6° for the G.E. Spectrophotometer in Fig. 8, 30° for the Taylor and Baumgartner instruments shown in Figs. 5 and 6, and 45° for the Color Eye shown in Fig. 9. This latter instrument and the Beckman Spectrophotometer actually reverse the geometric conditions of the other instruments of their respective types; that is, the Color Eye employs diffuse illumination 45° viewing, the Beckman instrument 0° illumination and 45° viewing. These interchanges of illuminating and viewing conditions have no effect on the instrument performances, however.

It will be noted that there are no X's opposite either the Beckman or Ansco 45° 0° instruments. The Beckman was included to show that spectrophotometers with both geometries are available. The Ansco can readily be adapted to the proper spectral functions by the development of new filters, but so far as the writer knows, such filters have not been developed. It may be noted in connection with the G.E. Spectrophotometer that it gives direct measurements of Y_C and Z_C when used with the new integrator that makes the total price of the apparatus about $14,000. Other spectral functions might be measured in the same automatic manner by the development of new cams for this integrator.

Lack of space and time prevents discussion of instrument features and details.

REFLECTANCE TEST PRACTICE IN FIELDS DEALING WITH WALLBOARD

The fields of paper and paint technology deal with the raw materials of fiberboard and with the manufacture of the finished product. The other technologists who deal with wallboard are illuminating engineers and architects who are concerned with end use of the product. Different practices in the measurement and specification of reflectance have grown up in these different technical fields.

Paper Technology

Paper brightness, or 45° 0° directional reflectance for blue light is widely used in the paper industry to measure the freedom of pulp, paper, clay, and other raw materials from blue-absorbing, yellowish impurities that limit the whiteness of the final product. The chief published methods of test are:

1. TAPPI Standard Method T 217 m-48, Brightness of Pulp ($R_{d\ (45°\ 0°)}$, B function of Fig. 3).
2. TAPPI Standard Method T 452 m-48, Brightness of Paper ($R_{d\ (45°\ 0°)}$, B function of Fig. 3).
3. ASTM Method of Test D 985 48 T, for 45° 0° Directional Reflectance for Blue Light (Brightness) of Paper (Tentative) ($R_{d\ (45°\ 0°)}$, Z_C function of Fig. 3).

Paint Technology

Those who deal with paint materials deal with surfaces that have gloss, as well as with those that do not. Where gloss is involved, there may be significant differences between R_t and $R_{d\ (45°\ 0°)}$. The paint technologist thinks of gloss and the specularly reflected light which is responsible, as being separate from the diffuse reflectance and color of painted surfaces. For "reflectance" he therefore desires a measurement free of the effect of gloss. It is for this reason that he prefers

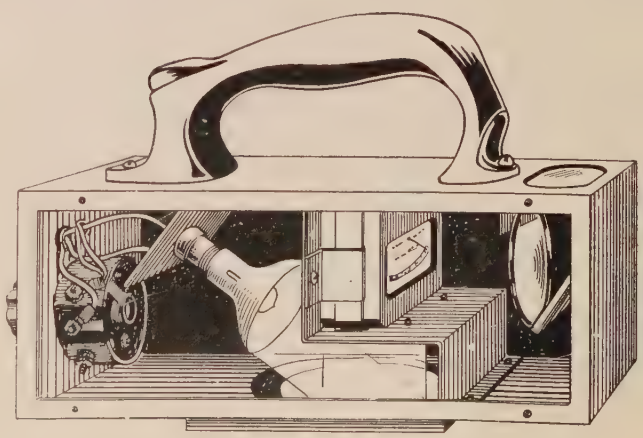

Fig. 13. Gardner 45° 0° Portable Reflectometer

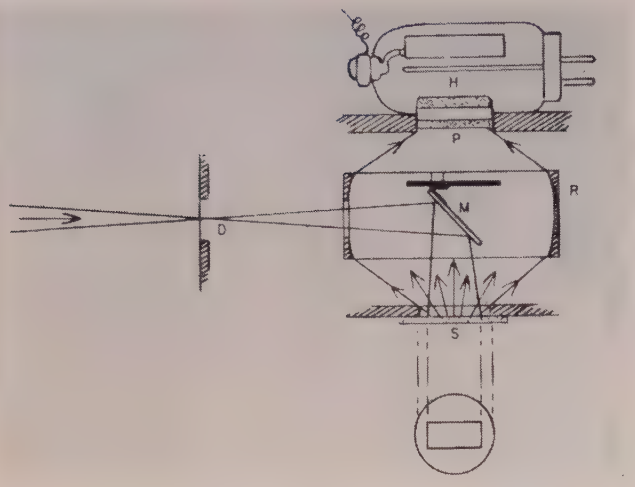

Fig. 14. Reflectance attachment Beckman Model DU Spectrophotometer

$R_{d\ (45°\,0°)}$ for his measurements of reflectance. Ordinarily, luminous *daylight* directional reflectance is used, since to the paint technologist, north sky *daylight* is the standard illuminant for color matching. Published tests for 45° 0° daylight luminous directional reflectance which are frequently used are:

1. ASTM Method of Test D 771–47 for Daylight 45° 0° Luminous Directional Reflectance of Paint Finishes ($R_{d\ (45°\,0°)}$, Y_C function of Fig. 3).
2. Method 612.1, Directional Reflectance, of Federal Specification TT-P-141b, Methods of . . . Testing Paint, . . . and Related Materials ($R_{d\ (45°\,0°)}$, Y_C function of Fig. 3).

Illuminating Engineering and Architecture

There seem to be no published standard methods of reflectance measurement in these fields, although definitions and practice have become well standardized in the field on the procedures established by Taylor when he developed his $R_{t\ (30°)}$ instrument for luminous lamplight reflectance 30 years ago (*11*). This instrument, and the Baumgartner reflectometer (*2*) designed to duplicate it photoelectrically, give the illuminating engineer the data he desires, because they tell him how much of the light falling on a surface is conserved. The illuminating engineer thinks of the wall of a room as a component of an indirect lighting system. To him, reflectance is a measure of the efficiency of this component. Since lighting installations usually involve light nearer to the color of lamplight than daylight, total luminous reflectance for incandescent light

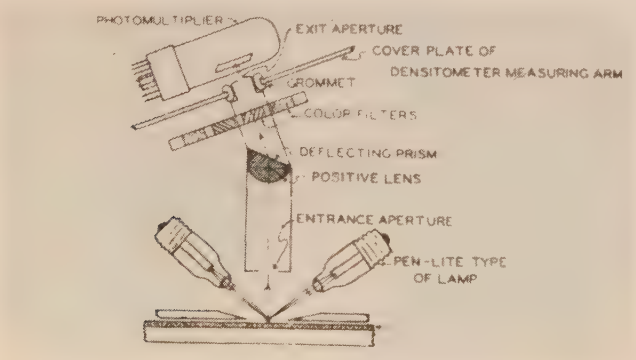

Fig. 15. Reflection attachment for Ansco (Scout) color densitometer

is the quantity of primary interest in this field. The architect customarily defers to the illuminating engineer when technical aspects of reflectance measurement are involved, and thus it is proper to say that reflectance is to the architect the same quantity as to the illuminating engineer.

RECOMMENDATIONS FOR REFLECTANCE TEST OF WALLBOARD

To establish a method of reflectance test for wallboard acceptable to each of the technical fields which deal with the product, it will be necessary to choose: (1) between total-reflectance methods employing diffuse collection of light and 45° 0° directional-reflectance methods, employing a single direction of view, and (2) between the blue-light "brightness" functions now used in the pulp and paper industry, and the luminosity functions covering the whole spectrum with maximum weight given the yellow and green.

It is shown in Table I that, of the two selection prob-

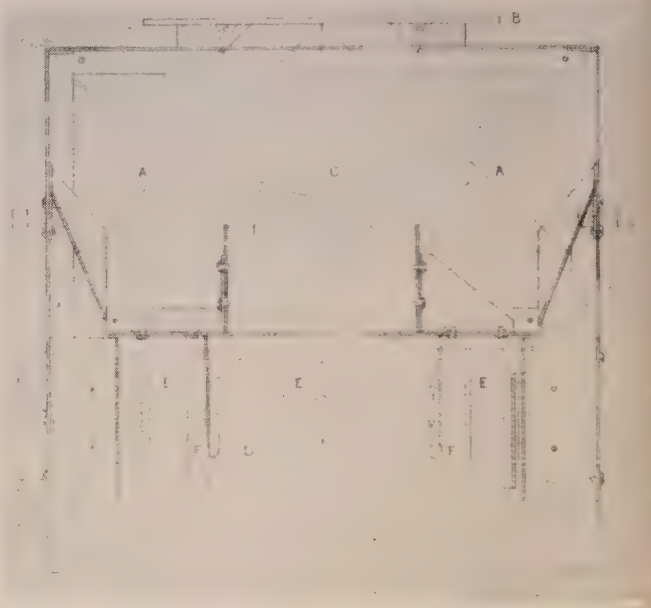

Fig. 16. Exposure head of 45° 0° Hunter Color-Difference Meter

lems, the second involving spectral functions, is much the more important so far as wallboard is concerned. To the writer there seems little justification for use of a blue-light "brightness" type of reflectance measurement in the wallboard field. There is no interest to users of wallboard in the lack of lignin or the ultimate whiteness obtainable in a fibrous wallboard by the use of blue dye. Paper is whitened with blue dye, but to give a wallboard good whiteness, paint is used and the wallboard takes the color and reflectance of the paint.

To measure and specify the reflectance of wallboard, the writer would therefore recommend some measure of luminous reflectance. The question of whether daylight or lamplight luminous reflectance should be measured, needs also to be decided, but this question is not critical. It would also be well to standardize on one geometry or the other. Although the above results show that it usually does not make much difference which geometry is used, it will be necessary to adopt either the present illuminating engineering practice with diffuse collection of reflected light, or the paint

and paper technology practice with unidirectional collection of reflected light. Considerations of instrument availability and cost, the present distribution of instruments, and the relative dominance of manufacturer and user of the product, will be involved in the final selection of a measurement method.

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Some Molecular Properties of the Hemicelluloses of Big Tooth Aspen

JAMES O. THOMPSON and LOUIS E. WISE

A study has been made of some molecular properties of representative hemicellulose fractions from big tooth aspen. The fractions were extracted with aqueous potassium hydroxide directly from the wood meal and also from a holocellulose prepared according to a modification of the Thomas procedure. Viscosity measurements on dilute solutions of these fractions in 10% aqueous potassium hydroxide and osmotic pressure measurements on chloroform solutions of a butyrate indicated a number average degree of polymerization of 155. This is in excellent agreement with the results reported by others for hemicelluloses isolated under mild conditions from wheat straw, beech, spruce, and paper birch. Aqueous solutions exhibited the typical electroviscous behavior of polyelectrolytes. Preliminary experiments, in which chain degradations were followed by viscosity measurements, showed that scissioning occurred in aqueous solution in the presence of alkali or of dilute mineral acid, and also on warming to about 60°C. or higher. Degradation in aqueous solution at 100°C. was marked and was accompanied by a significant decrease in pH.

The molecular weight and the molecular weight distribution are important physical properties of an isolated hemicellulose fraction. These properties are of particular interest and importance when the fraction has been isolated from the parent plant tissue with a minimum of degradation. The physical methods employed in measuring these properties in general are also excellent methods for following the course of chain degradation reactions. An extension of our present knowledge along these lines and especially in combination with extended chemical studies may be expected to improve markedly our understanding of the structure and behavior of these systems.

A review of the chemistry of the hemicelluloses has been presented by Wise (*1*). Detailed molecular size studies have been made by Husemann (*2*) on hemicellulose fractions isolated from wheat straw, beech, and spruce, and by Wethern (*3*) on fractions from black spruce. The number average degree of polymerization found for all these was essentially the same at about 155. Timell and Jahn (*4*) report a "very approximate value of 220 for the D.P." of a hemicellulose fraction from paper birch. This value was calculated from intrinsic viscosity measurements in cupriethylenediamine by assuming the same Staudinger constant as that shown by Husemann (*2*) to be valid for cuprammonium. In the present work it will be shown that this assumption is not justified and that their intrinsic viscosity when recalculated actually corresponds to a number average degree of polymerization of very nearly 155. Hemicellulose fractions from aspen were studied by means of a variety of physical methods by Millett and Stamm (*5*), who reported average degrees of polymerization ranging from about 60 to 80. The low yields of hemicelluloses isolated in this study, however, suggest that the fractions were somewhat degraded and, therefore, cannot be considered typical.

The chemical heterogeneity of aspen hemicelluloses has been studied by Thomas (*6*).

The present paper describes the results of an investigation in which physical methods have been employed to establish the degrees of polymerization of representative hemicellulose fractions isolated from big tooth aspen (*Populus grandidentata*). Some preliminary studies on chain degradation reactions are also described.

PHYSICAL METHODS

Viscosity

Viscosity measurements made with capillary viscometers on dilute solutions of high polymers can provide a rapid and accurate method for the determination of molecular weights (degrees of polymerization) (*7–9*). The method is not absolute, however, and calibration for each polymer-solvent system must be made by osmotic pressure, light scattering, or sedimentation

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equilibrium measurements. A relation between viscosity and molecular weight which has been found satisfactory for many polymer-solvent systems over extended ranges of molecular weight is of the form

$$[\eta] = KM^a \qquad (1)$$

where M is the molecular weight of the polymer, K and a are two constants which must be determined experimentally for each system by one of the absolute methods mentioned above, and $[\eta]$ is the intrinsic viscosity as defined by $\lim_{c \to 0} \eta_{sp.}/c$, or $\lim_{c \to 0} \ln \eta_r/c$, in which c is the concentration of the polymer (usually in grams per 100 ml. of solution), $\eta_{sp.}$ (specific viscosity) = (η solution − η solvent)/η solvent, and η_r (relative viscosity) = η solution/η solvent. The exponent a is associated with the degree of coiling of the long-chain polymer molecule in the given solvent. Its value depends on the nature of the polymer, as well as on the nature of the solvent and usually lies between 0.5 and 1.5. When the molecular weight range of interest is narrow—for example, from 15,000 to 30,000— equation (1) may be simplified to $[\eta] = K'M$ (2), where K' is a new constant which must be determined experimentally by means of an absolute method and which is valid only for the particular narrow range of molecular weight [unless, of course, the exponent a in equation (1) happens to be equal to 1]. This simplified equation has the advantage that only one absolute determination of M is required to establish the value of the constant K' for the particular system.

The intrinsic viscosity ($[\eta]$) of a given polymer in a given solvent is obtained experimentally by making viscosity measurements on a series of dilute solutions of different concentrations and plotting the reduced specific viscosity ($\eta_{sp.}/c$) against concentration (c). For most polymer-solvent systems, such a curve decreases linearly with decreasing concentration and the intercept obtained upon extrapolation to zero concentration represents the intrinsic viscosity. A typical plot of this type is represented by the lower curve in Fig. 3.

In the case of aqueous solutions of charged linear polymers—i.e., polyelectrolytes such as sodium pectinate, polyacrylic acid, and polyvinyl pyridine salts—the viscosity curves ($\eta_{sp.}/c$ versus c) are strongly concave upward (*10–12*) and extrapolation to infinite dilution does not yield a meaningful intercept (intrinsic viscosity). A typical plot of this type is represented by the upper curve in Fig. 3 Further, the addition of simple electrolytes to such solutions of polyelectrolytes has a marked effect on this anomalous electroviscous behavior. In general, the anomalous behavior may be suppressed completely by adding salt in relatively low concentration, e.g., 0.1 N and the reduced specific viscosity plot then becomes the typical straight line and a meaningful intercept ($[\eta]$) can be obtained.

The importance of this anomalous viscosity behavior in the present discussion lies in the fact that hemicellulose fractions, in general, are not soluble in organic solvents but are soluble in water (some fractions only at elevated temperatures) and that those fractions which are most readily soluble in water have a relatively high uronic acid content (*6*) and, hence, may be expected to behave as polyelectrolytes. Viscosity measurements on their aqueous solutions will then be capable of direct correlation with molecular weights only when the electroviscous effect has been completely suppressed.

Osmotic Pressure

Osmotic pressure measurements provide the simplest and most readily available method for determining absolute molecular weights of high polymers. A number of excellent discussions of this subject are available (*13–15*) and the general details will not be considered here. In essence, the method provides a means for counting the number of particles in a known weight of the high polymer in solution without regard to the size and shape of the individual particles. In the event that the particular polymeric system is not molecularly homogeneous, then the measurements yield a *number average* molecular weight (M_n). The more elaborate techniques of light scattering and sedimentation equilibrium are capable of yielding more detailed information than can be obtained from osmotic pressure measurements. They can, for instance, provide a measure of particle shape and of molecular size distribution. The osmotic pressure method, however, is adequate for the present purpose of calibrating the viscosity method of molecular weight determination. The precision of the method has been described by Spurlin and Elliot (*16*) as follows: "Over the range 25,000 to 100,000 the innate precision of the osmotic pressure method . . . is hardly approached by any other method of determination." Certain difficulties arise when dealing with aqueous solutions because of electric and other effects. These difficulties are markedly increased when the polymer is a polyelectrolyte (i.e., ionizable groups along the chain) and the degree of ionization is unknown, for, as indicated previously, each particle—whether long chain or simple ion—is counted equally by the osmotic method and is averaged into the M_n. In general, therefore, measurements are performed wherever possible in nonaqueous media and, in the case of hemicelluloses, for example, the preparation without degradation of derivatives soluble in organic solvents becomes of marked importance.

PREPARATION OF HEMICELLULOSES

The wood meal employed for this work was of 30 to 80 mesh and was prepared from a freshly cut and peeled big tooth aspen log by sawing and screening in the usual manner. It was extracted twice at room temperature with a 50% aqueous ethanol, filtered, and air dried prior to use. Two different methods were employed for isolating the hemicelluloses. The first involved the preparation of a holocellulose by a modification of the Thomas procedure (*6*) and the subsequent extraction with aqueous solutions of potassium hydroxide; the second method involved the direct extraction of the wood meal with aqueous potassium hydroxide.

Preliminary studies with a hemicellulose fraction isolated from a Thomas holocellulose with 5% potassium hydroxide solution indicated that appreciable chain degradation occurred (a) when aqueous solutions of the hemicellulose where heated to about 60°C.; (b) when aqueous solutions were acidified with dilute mineral acid at room temperature; and (c) when alkaline solutions were exposed to air. These factors were taken into account in designing the procedure to be used in isolating representative hemicellulose fractions

with a minimum of degradation. Wethern (*3*) prepared a series of holocelluloses from black spruce by a variety of methods and isolated corresponding hemicellulose fractions from each of these by alkaline extraction. On the basis of yields and dilute solution viscosities of these hemicellulose fractions, he concluded that a modified Thomas procedure yielded hemicelluloses fractions most suitable for molecular studies. Essentially this same method of holocellulose preparation was used in the present work. It was briefly as follows: Moist wood meal cooled to 0°C. was suspended in a saturated solution of chlorine in carbon tetrachloride at 0°C. and stirred vigorously for 10 minutes. The reaction vessel was cooled during this period so that the temperature of the reactants did not exceed about 5°C. The meal was then filtered on a Buchner funnel and given two washes with 3% ethanolamine in ethanol at room temperature. The solid was then removed from the Buchner funnel, extracted for 5 minutes with 3% alcoholic ethanolamine at room temperature, filtered, washed with ethanol at room temperature, with ice-cold ethanol and with ice-cold 50% aqueous ethanol, and then pressed dry on the filter. The color of the meal at this stage was a very dark red. The product was recooled and the chlorine-ethanolamine treatment was repeated twice. After the third treatment, the holocellulose was air dried. It was light tan in color and gave only a very faint Mäule reaction. It should be stressed that the primary object was not to obtain a lignin-free holocellulose but rather to obtain a holocellulose which would permit the isolation of the maximum yield of hemicelluloses with a minimum of degradation (such as might result from repeated chlorine treatments). It was felt that small quantities of lignin in the holocellulose would not interfere with the subsequent isolation of representative hemicellulose fractions. The yield of holocellulose obtained was 86% (oven-dry basis), indicating that lignin was still present.

Hemicellulose fractions were extracted from the holocellulose and from the wood meal by the same procedure. Successive extractions with 5 and 16% aqueous potassium hydroxide for periods of 1 hour at room temperature in an atmosphere of purified nitrogen yielded two fractions; these hemicellulose fractions were designated the "5% extract" and the "16% extract," respectively. The solid hemicelluloses were recovered from the caustic solutions by pouring the latter into 4 volumes of ethanol containing sufficient acetic acid to adjust the pH to approximately 6.5. The flocculent precipitate was centrifuged, washed several times with 85% aqueous ethanol, absolute ethanol, and absolute ether and then dried in a vacuum at room temperature. The yields—corrected for moisture and ash—were expressed as a percentage of the oven-dry extractive-free wood. Intrinsic viscosities of these fractions in 10% aqueous potassium hydroxide at 30°C. were determined by using Ostwald-Fenske viscometers which had flow times of about 200 seconds for water. Data obtained for the four fractions are summarized in Table I.

It is interesting to note that the total yield of hemicelluloses isolated by direct extraction from the wood meal was very nearly 85% of that obtained from the holocellulose; although the former gave somewhat stronger Mäule reactions, they were white in color rather than light tan as were the latter. The higher intrinsic viscosities obtained for the fractions isolated directly from the wood suggest that some chain degradation occurred during the preparation of the holocellulose. If it is

Table I. Comparative Data for Hemicellulose Fractions Isolated from Holocellulose and from Wood Meal

Fraction	*Color*	*Yield,*[a] *%*	$[\eta]^b$	*Uronic anhydride,*[b] *%*	*Mäule test*
		Holocellulose			
5% extract	Light tan	18.3	0.60	14.8	Faint (trace)
16% extract	Light tan	5.2	0.69	11.5	Faint (trace)
	Total	23.5			
		Wood Meal[c, d]			
5% extract	White	14.0	0.65	..	Trace[c]
16% extract	White	5.7	0.75	..	Trace[c]
	Total	19.7			

[a] Expressed as percentage of extractive-free wood and corrected for moisture and ash.
[b] Corrected for moisture and ash.
[c] Similar fractions isolated directly from trembling aspen wood (*Populus tremuloides*) meal though obtained in somewhat lower yield showed essentially the same intrinsic viscosities.
[d] Corresponding roughly to a few tenths of 1% lignin.

assumed that this degradation involves a random scissioning process, the extent of the degradation (i.e., the number of scissions per chain) is slight. If, however, the degradation involves preferential scissioning of groups from the ends of the chains, the number of scissions will be very much greater (*17*).

DEGRADATION OF HEMICELLULOSES

The fractions isolated with 5% potassium hydroxide were partially soluble in cold water and became completely soluble on warming. Experiments were performed to determine whether solution on warming was accompanied by chain degradation. A 0.5% solution of the hemicelluloses in water was prepared

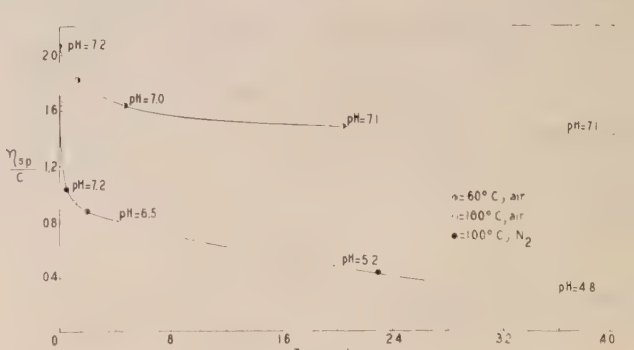

Fig. 1. Plot of viscosity against time showing the degradative influence of heat at 60 and 100°C. on a hemicellulose fraction in water

with the minimum of heating; it was then cooled to room temperature and its specific viscosity and pH determined. Portions of this solution were sealed in glass ampoules in the presence of air and heated for various times at 60 and 100°C. Viscosity and pH measurements were then made at the end of each of these periods. A plot of the reduced viscosity (η_{sp}/c) against time (t) for the two temperatures is shown in Fig. 1. The 100°C. experiment was also performed in an atmos-

phere of nitrogen; the corresponding plot was found to be identical with that for the product heated in the presence of air. The data indicate clearly that the hemicelluloses in aqueous solution are degraded upon heating and that the severity of this degradation increases as the temperature is increased. It is interesting to note that heating at 60°C. for 74 hours caused less reduction in viscosity than did 1 hour at 100°C., and that the pH's of these two reaction mixtures were essentially the same as that of the original solution. Of further considerable interest is the fact that, in the 100°C. experiment, the pH was constant during the initial sharp drop in viscosity, whereas it fell off quite markedly as heating was continued; this was accompanied by a somewhat diminished rate of viscosity decline. This lower rate of viscosity decline corresponds approximately to what would be expected for a uniform rate of random scissioning (*17*). It seems certain, on the basis of this preliminary work, that some degradative reaction occurred at 100°C. which did not occur at an appreciable rate at 60°C. A more detailed study will be required before an accurate interpretation of these interesting observations can be made. Heating of the dry hemicellulose in air at 100°C. for 120 hours produced no detectable chain degradation, as indicated by viscosity measurements.

The degradative influence of alkali and air at 30°C. on hemicellulose chain lengths is shown in Fig. 2, in which the reduced viscosity ($\eta_{sp.}/c$) for a 0.8% solution of a 5% extract hemicellulose in 10% aqueous potassium hydroxide is plotted against time. The reaction vessel employed was simply the capillary viscometer in which the initial viscosity was measured. The data were obtained by maintaining the viscometer (exposed to

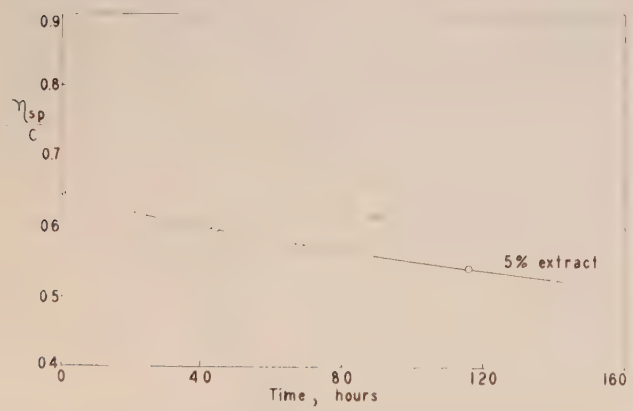

Fig. 2. Plot of viscosity against time showing the degradative influence of alkali at room temperature and in the presence of air on a hemicellulose fraction

air) in the water bath at 30°C., and repeating the viscosity measurements at intervals. A similar behavior was reported for black spruce hemicelluloses by Wethern (*3*), who found also that, even in the absence of air, significant degradation occurred over prolonged periods.

The rapid degradation of hemicellulose chains by very dilute mineral acids at room temperature was suggested by the results of the following experiment. An aqueous 5% potassium hydroxide extract of the holocellulose was divided into two equal parts; the first was poured in the usual manner into 4 volumes of ethanol containing acetic acid to adjust the pH to about 6, and the second was poured into alcohol acidified with hydrochloric acid to adjust the pH to about 3.5. The two hemicelluloses were then isolated and dried in the manner previously described. The reduced specific viscosities of 0.5% solutions of these two materials in water were 1.3 and 0.9 and their pH's were 6 and 3.5, respectively. These pH values indicate that, in the first of these fractions, the uronic acids were present largely as potassium salts, whereas in the second fraction they were largely free. The lower viscosity for the material at the lower pH suggests that chain degradation had resulted from contact with the dilute mineral acid. A clear-cut interpretation of these data, however, is not possible at this stage, for it is possible that the polyelectrolyte character of the hemicelluloses—quite apart from any possible chain degradation—may account in large part for the results obtained.

FRACTIONATION OF HEMICELLULOSES

Aspen hemicelluloses are known to contain relatively high percentages of uronic acid groups and, further, these percentages are known to be higher in the fractions more readily soluble in water (*6*). Analyses of the 5% extract and the 16% extract for uronic anhydride by the method of Browning (*18*) gave 14.8 and 11.5%, respectively (corresponding to one carboxyl group for each nine and eleven pentose units). In order to determine whether the hemicelluloses of aspen exhibit an appreciable polyelectrolyte character, a rough fractionation of a 5% extract hemicellulose from the holocellulose was performed. The moist hemicellulose, isolated by precipitation with alcohol and washed with 85% ethanol as previously described, was covered with distilled water (60 ml. per gram of air-dry hemicellulose) and stirred vigorously at room temperature for 2 hours. The water-soluble portion was separated by means of a centrifuge and was isolated from solution by precipitation with excess alcohol in the manner previously described, with the exception that no acetic acid was added to the alcohol. The solid from the centrifuge was re-extracted with water to yield a second water-soluble fraction. In the latter case, it is interesting to note that the solid hemicelluloses did not precipitate on pouring the water solution into excess ethanol until a trace of electrolyte—potassium acetate in this case—was added. A third fraction was obtained by extraction of the residue at room temperature with a 1% solution of potassium hydroxide, and a fourth by solution of the greater portion of the residue in 5% potassium hydroxide. The last two fractions were isolated from solution by pouring into excess ethanol which contained sufficient acetic acid to neutralize the alkali. They were then worked up as previously described. Data obtained for the four fractions are summarized in Table II. It is interesting to note that the light tan color of the parent hemicellulose was present almost exclusively in the water-soluble fractions.

It must be stressed that the fractionation performed was rough and was intended only to determine whether marked differences existed between the intrinsic viscosities of the more readily and the less readily soluble portions, and to provide a water-soluble fraction suitable for polyelectrolyte studies. It is clear from the data in Table II that significant differences do exist between

he intrinsic viscosities of the fractions. It is possible hat these differences may result from chemical differences of the type demonstrated by Thomas (*6*) for similar fractions, but it seems more probable that chain length differences are largely responsible. Absolute

Table II. Fractionation of the Hemicellulose (5% Extract)

Fraction	*Solvent*	*Yield,*[a] *%*	$[\eta]$[b]	*Uronic anhydride,*[c] *%*
FAA	Water	40	0.57	20.0
FAB	Water	25	0.64	..
FAC	1% KOH	20	0.68	..
FAD	5% KOH	15	0.71	..

[a] Based on estimated weight of parent hemicellulose.
[b] 10% aqueous potassium hydroxide as solvent (identical results were obtained with 6% aqueous sodium hydroxide as solvent); results corrected for moisture and ash.
[c] Corrected for moisture and ash.

molecular weight determinations on these fractions (or on their undegraded derivatives) would be required before a definite conclusion could be reached. Marked experimental difficulties must be overcome before either approach can be successfully pursued.

POLYELECTROLYTE BEHAVIOR

The dilute solution viscosity of the water-soluble fraction FAA in distilled water was measured over a range of concentrations to determine whether a significant polyelectrolyte behavior could be detected. This fraction contained 20% uronic anhydride, which corresponds to one carboxyl group for each six pentose sugar units. Particular care was taken with the viscosity measurements to ensure precision on the more dilute solutions. A plot of the reduced specific viscosity ($\eta_{sp.}/c$,) against concentration is represented by the upper curve of Fig. 3. The increase in reduced viscosity as concentration decreases is typical polyelectro-

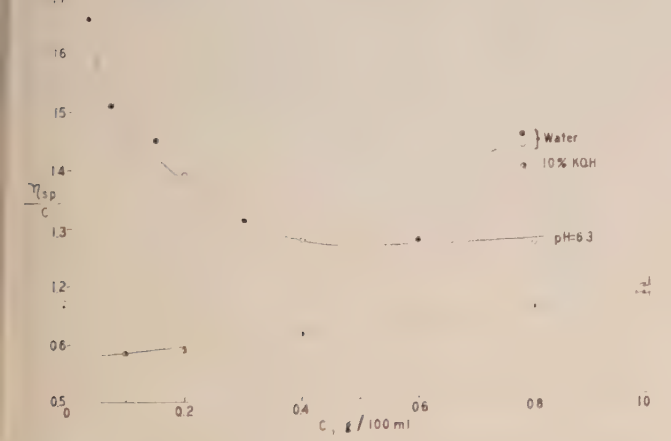

Fig. 3. Plot of viscosity against concentration for aspen hemicellulose fraction FAA in water and in 10% potassium hydroxide solution

lyte behavior and may be explained on the basis of the presence of negatively charged groups (ionized carboxyl) along the polymer chains. In the more concentrated solutions these negative charges are masked to a large extent by the presence of the ions from the other polymer chains and each chain is then free to assume a more or less randomly coiled configuration. As the concentration of the polymer (and, hence, of the ions) is reduced, the mutually repulsive influence of the negative charges along the chains becomes more marked and the polymer chains are forced to assume more extended (less coiled) configurations. In this more extended state, each polymer molecule contributes more to the viscosity of the solution than it did when in the more coiled configuration; hence, the reduced specific viscosity increases as the concentration of the polyelectrolyte decreases. It is clear from the shape of the upper curve in Fig. 3 that extrapolation to zero concentration to yield a meaningful intrinsic viscosity is impossible.

Low concentrations of simple electrolytes suffice to eliminate this anomalous polyelectrolyte effect. In the present study, however, 10% aqueous potassium hydroxide was employed because it served also as a solvent for the hemicellulose fractions which were not soluble in cold water; this permitted a comparison of the intrinsic viscosities of all the fractions in a single solvent. The typical $\eta_{sp.}/c$ versus c plot is represented by the lower curve in Fig. 3 which is that for fraction FAA in 10% aqueous potassium hydroxide. A similar, though less pronounced, polyelectrolyte effect was observed with fraction FAB in distilled water. The reduced effect probably resulted from a lower concentration of carboxyls along the polymer chains.

MOLECULAR WEIGHTS

Osmotic pressure measurements on chloroform solutions of a butyrate prepared from the water-soluble hemicellulose fraction FAA provided an absolute molecular weight determination of the hemicellulose fraction of lowest intrinsic viscosity. Butyrylation by the method of Carson and Maclay (*19*) was shown by Wethern (*3*) to produce almost no chain degradation when applied to the hemicelluloses of black spruce. In the present work a single butyrylation was employed so as to ensure minimum degradation, and the yield of derivative based on the weight of hemicellulose esterified was 180%. Seventy-two per cent of the product was soluble in chloroform and the degree of substitution of the soluble portion as determined by the method of Genung and Mallatt (*20*) was 2.0 (calculated assuming a pentosan). Osmometers of the Zimm and Myerson design were employed and the membranes were prepared from Du Pont 600 PT nonwaterproofed cellophane by swelling in 12% aqueous potassium hydroxide at room temperature for 30 minutes. The period of half life of the osmometer for chloroform at 30°C. was one hour; and the semistatic method of measurement was employed.

The number average molecular weight of the derivative was found to be 35,000 which corresponds to a number average molecular weight for the hemicellulose of 17,000. The intrinsic viscosity of the derivative in chloroform at 30°C. was 0.54.

This molecular weight of 17,000 for the hemicellulose corresponds to a number average degree of polymerization (D.P.) of 130 (calculated as a pentosan). If this value is taken as the average for hemicellulose fraction FAA, then a correlation between D.P. and intrinsic viscosity in aqueous 10% potassium hydroxide may be made by using the relation $[\eta] = K'$ (D.P.). The experimental data thus give a value for K' of 4.4×10^{-3}

which is in good agreement with the corresponding values reported by Husemann(*2*) for the hemicelluloses of spruce, beech, and straw, and also with the results of Wethern (*3*) for black spruce. The value of the constant K' thus obtained may be applied with reasonable confidence to estimate the number average degrees of polymerization of the other hemicellulose fractions. The values thus calculated are shown in Table III.

Table III. Molecular Weights of the Hemicellulose Fractions

Fraction	$[\eta]$	*Molecular weight*[a]	*Degree of polymerization*[a, b]
Holocellulose			
5% extract	0.60	18,000	140
16% extract	0.69	21,000	160
Wood Meal			
5% extract	0.65	19,000	150
16% extract	0.75	22,000	170
5% Extract from Holocellulose			
FAA	0.57	17,000	130
FAB	0.64	19,000	140
FAC	0.68	20,000	150
FAD	0.71	21,000	160

[a] Number average.
[b] Calculated as pentosan.

Of marked interest also is the fact that these results are almost identical with those found in more detailed molecular size studies by Husemann (*2*) for the hemicelluloses of beech, straw, and spruce, and by Wethern (*3*) for black spruce. The fractionation method employed in the present study suggests a narrow size distribution for the aspen hemicelluloses but more detailed studies must be made before definite conclusions can be drawn.

INTRINSIC VISCOSITIES IN CUPRIETHYLENEDIAMINE AND IN CUPRAMMONIUM HYDROXIDE

The intrinsic viscosities of the 5% extract and the 16% extract hemicellulose fractions from the holocellulose were measured in cupriethylenediamine and in cuprammonium hydroxide. The compositions of the two solvents were those normally employed in the TAPPI viscosity procedures (*21*). The results obtained are shown in Table IV, together with those previously

Table IV. Intrinsic Viscosities in Different Solvents

Hemicellulose fraction	*Intrinsic viscosity in*— 10% *Potassium hydroxide*	*Cuprammonium hydroxide*	*Cupriethylenediamine*
5% extract	0.60	0.63	0.90
16% extract	0.69	0.81	1.14

obtained with 10% aqueous potassium hydroxide as solvent. It will be seen that the intrinsic viscosities of the 5% extract in aqueous potassium hydroxide and in cuprammonium hydroxide are very nearly equal—that for cuprammonium being slightly the larger. This is in agreement with the findings of Husemann (*2*) for hemicelluloses from wheat straw, beech, and spruce. The intrinsic viscosity of the 16% extract in cuprammonium, however, is significantly greater than that in aqueous potassium hydroxide. A comparison of the intrinsic viscosities in cuprammonium hydroxide and in cupriethylenediamine shows that, in the case of both fractions, the viscosity in cupriethylenediamine is slightly greater than 1.4 times that in cuprammonium hydroxide. This is in excellent agreement with the average value of 1.365 reported by Conrad, et al. (*22*) for a series of celluloses covering a wide range of molecular weights.

The "very approximate value of 220 for the D.P." of paper birch hemicellulose reported by Timell and Jahn (*4*) was calculated from the intrinsic viscosity in cupriethylenediamine by using the Staudinger constant "usually assumed to be valid for cuprammonium solutions of cellulose." Husemann (*2*) has shown that the Staudinger constant relating intrinsic viscosity and number average D.P. is essentially the same for hemicelluloses and cellulose in cuprammonium hydroxide. The experimental evidence clearly indicates, however, that this same constant may not be used for cupriethylenediamine solutions, and it would appear that a more nearly correct value for the number average degree of polymerization of paper birch would be 155. This recalculated value is in excellent agreement with the values obtained for the hemicelluloses isolated under mild conditions by other workers from wheat straw, beech, spruce, and aspen.

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ASSOCIATION NEWS AND EVENTS

Report of Nominating Committee

The TAPPI 1953 Nominating Committee, consisting of Roderick O'Donoghue, Consulting Engineer, New York, N. Y., A. E. H. Fair, Alliance Paper Mills, Merritton, Ont., John J. Goss, Gaylord Container Corp., Bogalusa, La., Frederick S. Klein, Byron Weston Co., and Kenneth J. Mackenzie, Eastman Kodak Co., Rochester, N. Y., have presented the following nominees for officers and executive committeemen to be voted on by the Association membership prior to the TAPPI Annual Meeting in 1953:

President (one year): George H. Pringle, Mead Corp., Chillicothe, Ohio

Vice-President (one year): Karl O. Elderkin, Bowaters Southern Paper Corp., Greenville, S. C.

Executive Committeemen (three years): Norman I. Bearse, Champion-International Corp., Lawrence, Mass.; John Buss, Provincial Paper, Ltd., Toronto, Ont.; Harry F. Moore, Beloit Iron Works, Beloit, Wis.; Charles J. Sibler, West Virginia Pulp & Paper Co., New York, N. Y.

Note: Article VII, Sec. 3, TAPPI Constitution: "Other nominations, if signed by at least 25 voting members, may be submitted to the Secretary of the Association, in writing, on or before November 20, and if each nomination so made is accepted in writing by the nominee, the name shall be placed on the official ballot."

Testing Conference

The TAPPI Testing Division will sponsor a Conference on Testing Methods on Sept. 10–12, 1952, to be held at the College of Forestry, State University of New York, Syracuse, N. Y. Sessions will be held by the Nonfibrous Materials Testing Committee, Wax Testing Committee, Pulp Testing Committee, Optical Properties Committee, Microscopy Committee, Paper Testing Committee, Packaging Materials Testing Committee, and the Chemical Methods Committee. All members of TAPPI Testing Division committees are urged to advise their committee chairmen if they can be present. Any member of the Technical Association is welcome to attend the Conference and should advise R. G. Macdonald, Secretary of TAPPI, 122 E. 42nd St., New York 17, N. Y., if he desires to attend. This information is needed to assure adequate meeting room capacity. Hotel reservations should be made as soon as possible at either the Onondaga Hotel or the Hotel Syracuse. No sessions will be held at these hotels.

Certificate of Merit

For the second successive year in its three years of existence *Tappi* received a major award in *Industrial Marketing*'s 14th Editorial Achievement Competition. A Certificate of Merit was awarded for graphic presentation and format of its technical articles.

Committee Secretary

H. O. Teeple of the International Nickel Co., New York, N. Y., has been appointed Secretary of the TAPPI Chemical Engineering Committee and the Digester Corrosion Subcommittee by James R. Lientz, Union Bag & Paper Corp., Savannah, Ga., Chairman.

Alkaline Pulping Conference

Fred W. Bishop, Chief Chemist of Southland Paper Mills, Inc., Lufkin, Tex., has been appointed Program Committee Chairman of the TAPPI Alkaline Pulping Conference to be held at the Battle House, Mobile, Ala., on Nov. 12–14, 1952. The program will feature kraft mill recovery problems and a general program dealing with alkaline pulping subjects of current interest. Hotel reservations should be made direct with the Battle House. In making reservations please mention the TAPPI Alkaline Pulping Conference. Official tours will be made through the mills of the International Paper Co., Southern Kraft Division, and Hollingsworth & Whitney Co. in Mobile.

Fifty-Two Hundred TAPPI Members

As of June 15 the membership of the Technical Association was 5200. This total is expected to increase somewhat before the end of the year. This is a new high in TAPPI membership.

Pacific Seminar

The TAPPI Pacific Section will hold another seminar this Fall. Professor Karl Freudenberg, of Heidelberg University Chemical Institute, Heidelberg, Germany, will be the lecturer.

Lectures will be given at Portland, Ore., on September 18 and 19, and in Seattle, Wash., on September 22 and 23. These lectures will deal with "Recent Research on Cellulose and Lignin." Separate lectures will be entitled: (1) "Organic Chemical Work on the Constitution of Cellulose," (2) "Research on Natural Lignin and Its Constitution," (3) "Research on Artificial Lignin and Its Constitution," and (4) "Lignification of Plant Tissue, and the Lignin-Polysaccharide Linkage."

It is possible that Dr. Freudenberg will be able to speak at other Section meetings.

The Fall meeting of the Pacific Section will be held at Victoria, B. C., on September 26 and 27. This meeting will be held jointly with the Western Branch of the CPPA Technical Section.

Fibrous Agricultural Residues Conference

The TAPPI Fibrous Agricultural Residues Committee will sponsor its ninth national conference at the Northern Regional Research Laboratory, Peoria, Ill., on Oct. 27–28, 1952. S. I. Aronovsky is Chairman of the Committee. The conference will feature problems in "Straw Specifications and Grading."

Hotel room reservations should be made direct with the Pere Marquette Hotel in Peoria.

Plastics Conference

The TAPPI Plastics Committee, E. C. Jahn, Chairman, will sponsor a Plastics Conference at the College of Forestry, State University of New York, Syracuse, N. Y., on Nov. 5–6, 1952. The program of November 5 will be devoted to "New Resin Developments and Their Possible Applications to

Paper and Paper Products." Professor Herman Mark of Polytechnic Institute of Brooklyn will be Chairman.

The program of November 6 will feature "Application of Resins in Paper Converting." Ralph T. Nazzaro of the Westfield River Paper Co. will be Chairman of these sessions.

Papers will be presented at the morning sessions. The afternoon sessions will feature panel discussions of the papers presented in the mornings.

Hotel room reservations should be made early at the Hotel Syracuse or the Onondaga Hotel in Syracuse.

Corrugated Container Conference

The TAPPI Corrugated Container Committee, F. D. Long, Container Corp. of America, Chicago, Ill., Chairman, will sponsor a Corrugated Container Conference at the Schroeder Hotel, Milwaukee, Wis., on Nov. 20–21, 1952. One day will be devoted to visits to corrugated container plants and the other to the discussion of prepared presentations.

The committee arranging the meeting program includes Burt Mendlin, Cornell Paperboard Products Co., Milwaukee, Wis., J. J. Koenig, Gaylord Container Corp., St. Louis, Mo., G. G. Johnston, Waldorf Paper Products Co., St. Paul, Minn., W. R. Tobin, American Box Board Co., Grand Rapids, Mich., H. T. Scordis, Union Bag & Paper Corp., Savannah, Ga., and F. D. Long, Container Corp. of America, Chicago, Ill.

Hotel reservations should be made direct with the Schroeder Hotel, Milwaukee, Wis. The TAPPI Corrugated Container Conference should be mentioned in making reservations.

Seventh Engineering Conference

An excellent program has been prepared for the Seventh Engineering Conference of the Pulp and Paper Industry, sponsored by the TAPPI Engineering Division and to be held at the Morrison Hotel, Chicago, Ill., on Oct. 13–16, 1952. Charles J. Sibler, Chief Engineer of the West Virginia Pulp & Paper Co., New York, N. Y., is General Chairman of the Engineering Division.

Sessions will be held by the Steam and Power, Materials Handling, Drying and Ventilating, Engineering Research and Machine Design, Mill Design and Economic Aspect, Hydraulics, Mill Maintenance and Materials, Electrical Engineering, and Chemical Engineering Committees. A tentative program is given below.

Visits will be made to the plants of the Link-Belt Co. and Crane Co. in Chicago.

Hotel reservations should be made direct with the Morrison Hotel. The TAPPI Engineering Conference should be mentioned.

Program of Monday, October 13

8:30 a.m. Registration

9:15 a.m. General Session

C. J. Sibler, West Virginia Pulp & Paper Co., New York, N. Y., *Chairman*

Addresses of Welcome

9:30 a.m. Materials Handling Session

A. P. Schnyder, Ebasco Services, New York, N. Y., *Chairman*

A. "Pulpwood Felling, Preparation and Transportation from the Forest," by James E. Holekamp, Logging Engineer, American Pulpwood Assoc., New York, N. Y.

11:15 a.m. Mill Maintenance and Materials Session

H. F. Parker, New York and Pennsylvania Co., Lock Haven, Pa., *Chairman*

A. "Engineer's Approach to Safety," by George Harper, University of Illinois, Urbana, Ill.

B. "Care and Maintenance of Rubber Covered Rolls in the Pulp and Paper Industry," by representative of the United States Rubber Co., New York, N. Y.

12:30 a.m. Luncheon

Engineering Division Committee Luncheons

2:00 p.m. Drying and Ventilating Session

M. L. Barker, Beckett Paper Co., Hamilton, Ohio, *Chairman*

A. "Increasing Drying Capacity Without Increasing Over-all Machine Dimensions," by W. H. Funk, Lukens Steel Co., Coatesville, Pa.

B. (Second topic later.)

3:30 p.m. Discussion Sessions

A. Materials Handling—"Harvesting and Handling of Smaller Sizes of Pulpwood from the Forest to the Mill Woodyard." Discussion Leader, W. A. McKenzie, Simpson Logging Co., Shelton, Wash.

B. Mill Maintenance—"Tool Room versus Store Room for Maintenance Tools"

C. Drying and Ventilating

6:30–8:30 p.m. Dinner (Where you will)

Program of Tuesday, October 14

9:00 a.m. Mill Design and Economic Aspects Session

Roland A. Packard, Mutual Security Administration, Washington, D. C., *Chairman*

A. "Paper Mill and Machine Reconstruction in Europe Since the War," by Arnold Barea, E. D. Jones & Sons, Pittsfield, Mass.

B. "Government and the Pulp and Paper Industry in the United States," by Allan Hyer, Bagley & Sewell Co., Watertown, N. Y.

10:30 a.m. Steam and Power Session

H. R. Arnold, Riegel Carolina Corp., Acme, N. C., *Chairman*

A. "The Combination of Hot Zeolite Softening of Boiler Feedwater," by J. D. Yoder, Permutit Co., New York, N. Y.

B. "Application of the Package Steam Generating Unit to the Paper Industry," by F. X. Gilig, The Babcock & Wilcox Co., New York, N. Y.

12:30 p.m. Luncheon

Engineering Division Committee Luncheons

2:00 p.m. Chemical Engineering Session

J. R. Lientz, Union Bag & Paper Corp., Savannah, Ga. *Chairman*

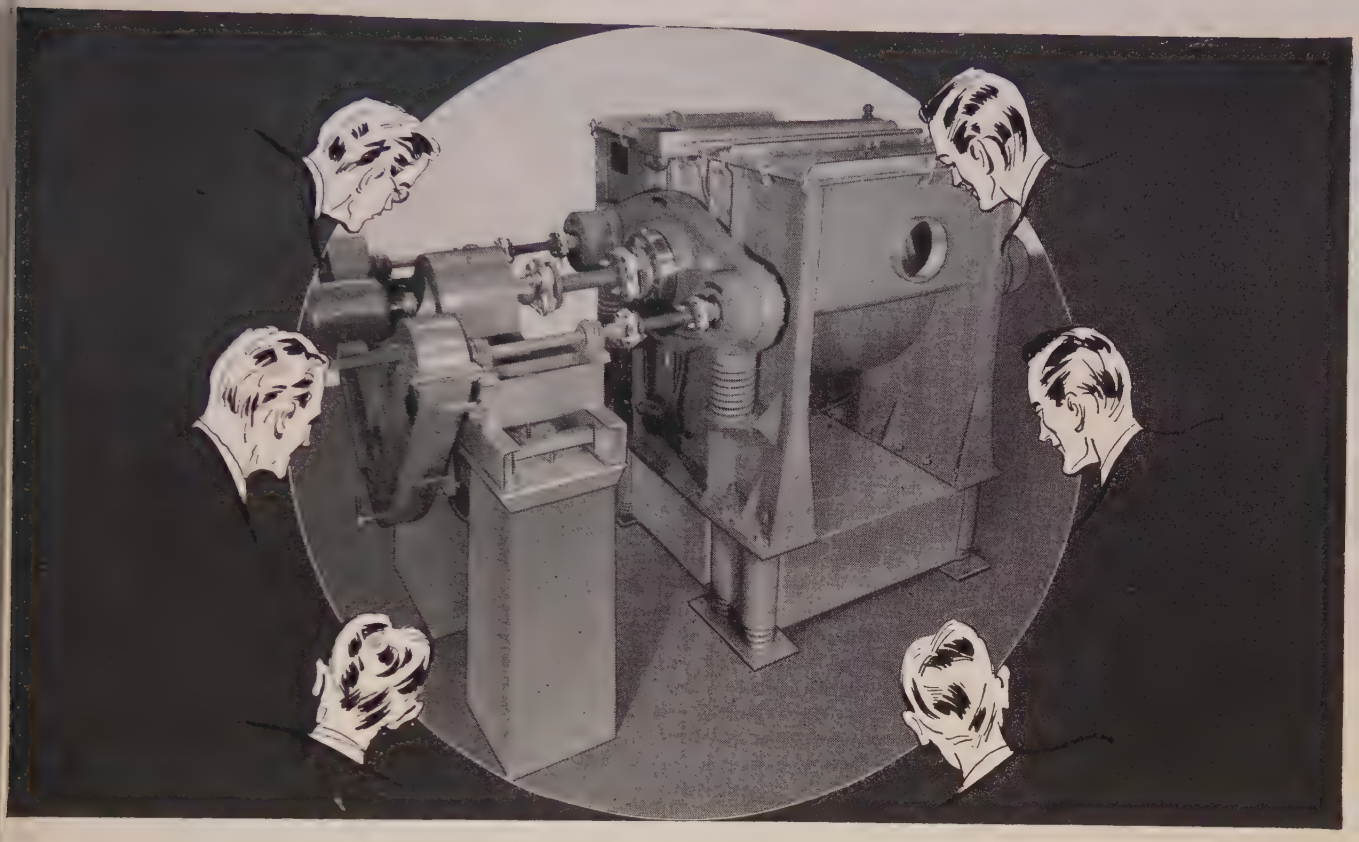
A. "Digester Corrosion Measurement," by Nicholas Shoumatoff, W. Va. Pulp & Paper Co., New York, N. Y.

B. Summary of Battelle Memorial Institute's special Report on "Analysis of Corrosion Questionnaires"

3:30 p.m. Discussion Sessions

A. Mill Design and Engineering Aspects (Committee Member Discussion)

Why This World Wide Interest in BIRD VIBROTOR SCREENS?



The answer is obvious — extraordinary performance

One mill is screening semi-chemical pulp at the rate of 50 to 60 tons per Vibrotor Screen per day — and has repeat ordered.

On unbleached kraft and sulphite stocks, Vibrotor Screens are handling up to 65 tons through .012″ cut plates with stock consistency at 1.6%.

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Ask us to tell you more about the Bird Vibrotor Screen and the remarkable results it can achieve in small space at low cost.

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B. Steam and Power—"Electrical Problems When Operating in Parallel with a Utility." Discussion Leader, D. F. Langenwalter, General Electric Co., Schenectady, N. Y.
C. Chemical Engineering—Discussion of Above and Discussion of Data Sheet on Digester Corrosion Inspection

7:00 p.m. Get-Together Party and Dinner (Sponsored by allied industries)

Program of Wednesday, October 15

9:00 a.m. Electrical Engineering Session

R. W. Foster, Champion Paper & Fiber Co., Canton, N. C., *Chairman,*

A. "Application of Electric Cables," a Project Report by A. O. Mortenson, St. Regis Paper Co., Cantonment, Fla.
B. "Power Requirements for Press Sections," a Project Report by R. R. Baker, Westinghouse Electric Corp., East Pittsburgh, Pa.

10:30 a.m. Hydraulics Session

K. J. Mackenzie, Eastman Kodak Co., Rochester N. Y., *Chairman*

A. "Correlation Between Shear Criterion and Pipe Friction Loss," a Project Report by R. E. Durst, University of Maine, Orono, Me.

12:30 p.m Engineering Division Luncheon

"People Have a Place in Your Planning," by A J. Miller, Jr., Assistant Director of Industrial Relations, Mead Corp., Chillicothe, Ohio

2:30 p.m. Engineering Research and Machine Design Session

J. D. Lyall, Armstrong Cork Co., Lancaster, Pa., *Chairman*

A. "Flow in Converging Nozzle-Type Slices." "Can Negative Pressures Be Present?" by J. B. Gough, Consultant, Mead Corp., Chillicothe, Ohio.
B. "A Method for Determining Sheet Making Rates in Cylinder Machine Operation," by Charles F. Payne, Eastman Kodak, Rochester, N. Y.
C. "An Analysis of a Cylinder Vat," by J. A. Jackson, St. Regis Kraft Co., Jacksonville, Fla.

3:45 p.m. Discussion Sessions

A. Electrical Engineering—"Power Requirements for Press Sections," Preliminary Report by J. Osborn, Bowaters Southern Paper Corp., Charleston, Tenn. Also: "Power Requirements of Supercalenders," by J. L. Van Nort, Reliance Electric & Engineering Co., Cleveland, Ohio
B. Hydraulics—Discussion of Morning Report
C. Engineering Research and Machine Design—Discussion of morning papers. Discussion of project on estimating costs of engineering projects

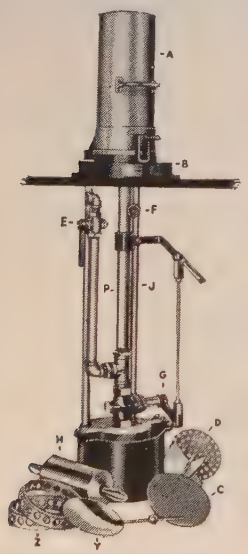
7:30 p.m. Dinner Meeting of Committee Chairmen and Secretaries

A. General Discussion
B. Data Sheets

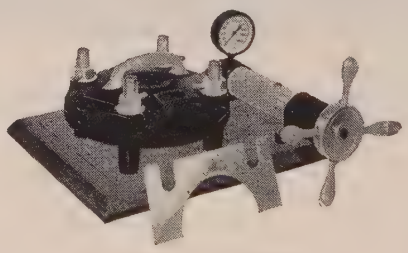
Program of Thursday, October 16

9:00 a.m. Mill Visits

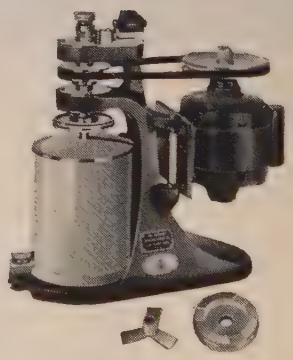
(Details later)



★ SHEET MACHINE
DRYER RINGS
AUXILIARY EQUIPMENT



★ PUMP AND PRESS



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Fibre Classification Grid—Used With Sheet Machine in Place of Standard Grid Plate

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HERMANN IMPROVED CLAFLIN REFINER
LANCASTER, OHIO

HERE'S PROVEN ON-THE-JOB PERFORMANCE WHERE CURLATORS ARE . . .

- Upgrading #2 sulphite pulp and screenings
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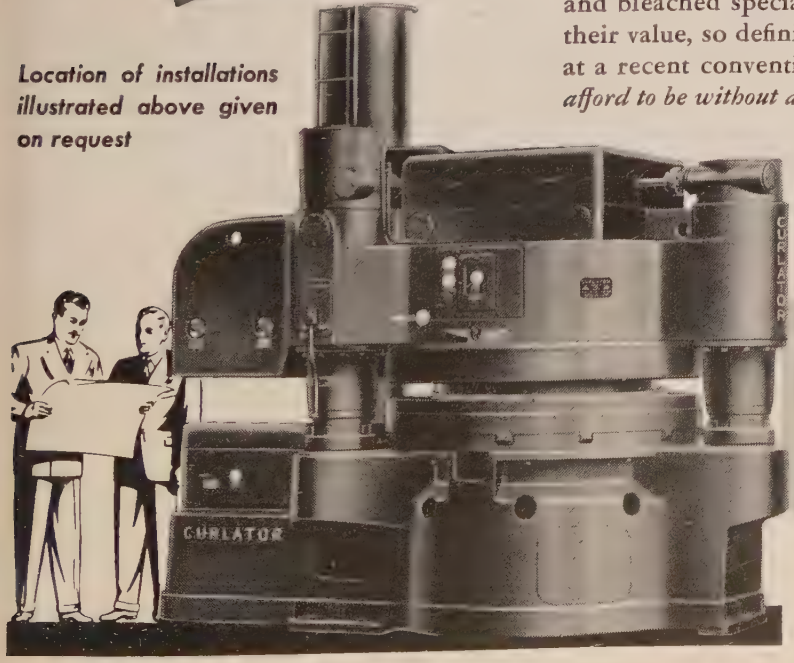
WRITE *today for detailed information.*

Location of installations illustrated above given on request



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PERSONAL MENTION

Items About New and Old Tappimen

New TAPPI Members

Carlos J. Benko, Technical Director, Companhia Santista de Papel, São Paulo, Brazil.

Roland J. E. Bilodeau, Research Chemist, Hollingsworth & Whitney Co., Inc., Winslow, Me., a 1950 graduate of the University of Maine.

Martin F. Bretl, Technical Representative, Rohm & Haas Co., Philadelphia, Pa., a 1936 graduate of the University of Wisconsin.

Kenneth J. Busse, Technical Adviser to Paper Mills, Glass Fibers, Inc., Toledo, Ohio, a 1949 graduate of the University of Wisconsin.

Ray M. Chermak, Electrical Engineer, Kalamazoo Vegetable Parchment Co., Parchment, Mich., a 1941 graduate of the University of Minnesota.

Manuel Firmino da Costa, Research Assistant, Laboratorio de Tecnologia de Celulose, Alcobaca, Portugal, a 1945 graduate of the Technical University of Lisbon.

Gennaro S. D'Addio, Salesman, Mead Sales Co., Chicago, Ill. Attended Northwestern University and Yale University.

Irwin M. Densen, Plant Manager, Eastern Corrugated Container Corp., Clifton, N. J., a 1946 graduate of New York University.

James Dillin, Vice-President, Industria, Inc., New York, N. Y.

Ingmar Kidem, Vice-President, Svenska Cellulose A.B., Sundsvall, Sweden, a 1933 graduate of the Royal Technical University of Stockholm.

Harold L. Field, Technical Supervisor, West Virginia Pulp & Paper Co., Mechanicville, N. Y., a 1942 graduate of New York State College of Forestry.

Julio Furth, Importer and Agent of Paper Making Machinery, Buenos Aires, Argentina, a 1920 graduate of the Technical College, University of Vienna, Austria.

Burton A. Gales, Chemist, Chicago Printed String, Chicago, Ill.

Esther B. Garber, Technical Librarian, Springdale Laboratories, Time, Inc., Springdale, Conn.

Martin N. Geib, Technologist, National Bureau of Standards, Washington, D. C., a 1920 graduate of Iowa University.

Richard P. Hamilton, Student, New York State College of Forestry, Syracuse, N. Y., a June, 1952 graduate.

Pralhad S. Hangal, Student, Pulp and Paper Dept., University of Maine, Orono, Me., a 1950 graduate of Benares Hindu University.

Md. Mozammel Haque, Student, Pulp and Paper Dept., University of Maine, Orono, Me., a 1946 graduate of Calcutta University.

Nils F. Janson, Lieutenant, Chief Engineer, U.S.S. *Noble*, San Francisco, Calif., a 1945 graduate of Rensselaer Polytechnic Institute.

Wayne E. Kendrik, Student, Pulp and Paper Technology, Western Michigan College, Kalamazoo, Mich., graduating in June 1952.

Fred A. Leser, Jr. Salesman, Samuel M. Langston Co., Camden, N. J., a 1947 graduate of Pennsylvania State College.

Douglas A. Leslie, Chief Chemist, Canada Glazed Papers Ltd., Toronto, Ont., Canada, a 1944 graduate of the University of Toronto.

Robert G. MacKay, Assistant Night Superintendent, Great Northern Paper Co., Millinocket, Me. Attended Massachusetts Institute of Technology.

A. Harold Martin, Technical Control Chemist, Falls Paper & Power Co., Oconto Falls, Wis., a 1944 graduate of the University of Wisconsin.

Ernest A. G. McLean, Chief Chemist, MacMillan & Bloedel (Alberni) Ltd., Pulp Div., Port Alberni, B. C., Canada, a 1948 graduate of the University of Alberta.

Arthur C. Megalos, Chemical Engineer, American Seal-Kap Corp., Long Island City, N. Y , a 1949 graduate of Brooklyn Polytechnic Institute.

Walter. P Molda, Technical Supervisor, Thilmany Pulp & Paper Co., Kaukauna, Wis., a 1941 graduate of Illinois Institute of Technology.

Arthur L. Noble, Mechanical Engineer, West Virginia Pulp and Paper Co., New York, N. Y., a 1947 graduate of New York University.

J. Luis Ochoa, Chemist, Interchemical Corp. de Mexico S. A., Mexico City, Mexico, a 1946 graduate of Universidad Nacl. Autonona de Mexico.

Charles W. Parsons, Assistant to Vice-President, Republic Flow Meters Co., Chicago, Ill., a 1920 graduate of Worcester Polytechnic Institute.

Stanley J. Pasternak, Assistant Superintendent, Hollingsworth & Vose Co., West Groton, Mass. Attended Massachusetts Institute of Technology.

Fleet C. Ratliff, Chemical Engineer, Gaylord Container Corp., Mill Div., Bogalusa, La., a 1934 graduate of Louisiana State University.

Jean A. Rebuffet, Chemist, Papeteries de France, Lancey (Isere), France, a 1947 graduate of Grenoble University.

Felix Ribot, Jr., Assistant Superintendent, Cartonera Moderna, S. A., Naucalpan, Estado de Mexico. Attended the Polytechnic College.

Walter B. Roberson, Research Chemist, Hudson Pulp & Paper Co., Palatka, Fla., a 1935 graduate of Arkansas State College.

H. Saevitzon, Director, Cellulose Products Ltd., Crown Mines, Johannesburg, S. A.

Edward D. Stanley, Director, Physical Testing Laboratories, Skinner and Sherman, Inc., Boston, Mass., a 1949 graduate of Tufts College.

Robert E. Strauss, Project Engineer, John Strange Paper Co., Menasha, Wis., a 1947 graduate of Purdue University.

Martin E. Swinford, Chief Chemist, Cupples Hesse Corp., St. Louis, Mo., a 1950 graduate of Washington University.

Harold W. Taylor, General Manager, Hercules Box Co., Columbus, Ohio, a 1921 graduate of Tufts College.

Phillip R. Templin, Chemist, Gulf Research & Development Co., Pittsburgh, Pa., a 1942 graduate of Susquehanna University.

John J. Turbyne, Beater Room Foreman, Hollingsworth & Whitney Co., Waterville, Me., a 1933 graduate of the University of Maine.

Ralph W. Turner, Engineer, Great Northern Paper Co., Millinocket, Me., a 1948 graduate of Massachusetts Institute of Technology.

Kyozo Uragi, President, The Kishu Paper & Pulp Mfg. Co., Ltd., Udono Village, Minamimurogun, Mie Prefecture, Japan, a 1941 graduate of Tohoku University.

Alan N. Vroom, Chief, Applied Physics Section, Weyerhaeuser Timber Co., Longview, Wash., a 1944 graduate of McGill University with a Ph.D degree.

Bruce L. Wally, Student, New York State College of Forestry, Syracuse, N. Y., a June, 1952 graduate.

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PARAFFIN
WARCO* 150 YELLOW
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Randall Wiggins, Technical Director, Boler Petroleum Co., Philadelphia, Pa., a 1938 graduate of the University of Pennsylvania.

TAPPI Notes

Claude M. Adams is now General Superintendent of the Southern Paperboard Co., Port Wentworth, N. J.

Earl F. Anderton, formerly of the Scott Paper Co., is now Plant Manager of the Coos Bay Pulp Corp., Empire, Ore.

Philip S. Barnhart, formerly of the Westfield River Paper Co., is now a Consultant at 20 Craiwell Ave., West Springfield, Mass.

Lee M. Bauer is now Production Manager of the Ecusta Paper Co., Pisgah Forest, N. C.

Carl J. Bergendahl, formerly of Pagel-Horton Co., is now Managing Director of Hafrestrons A/B, Havernd, Sweden.

Howard J. Blake has resigned as Research Director of the Wood Flong Corp., Hoosick Falls, N. Y. Mr. Blake's present address is P.O. Box 94, Hoosick Falls, N. Y.

Joseph E. Bradley, Jr., formerly of Watson-Park Co., is now Research Chemist for the Oxford Paper Co., Rumford, Me.

Earl O. Bryant is now Assistant Manager of Research and Development for the Ecusta Paper Co., Pisgah Forest, N. C.

Elmer R. Burling is now Executive Vice-President of the Improved Paper Machinery Co., Nashua, N. H.

John B. Canha, formerly of Chas. T. Main, Inc., is now Field Engineer for Pierce Consulting Engineering Co., Boston, Mass.

Edgar R. Carpenter, formerly of the Weston Paper & Mfg. Co., is now Senior Field Engineer for the B. F. Goodrich Co., Akron, Ohio.

Grellet M. Collins, Consultant, formerly of New York City, is now located at 407 Woodside Ave., Narberth, Pa.

Robert De S. Couch is now Special Assistant to the Division Manager, Carton & Container Div., General Foods Corp., Battle Creek, Mich.

Percy J. Cox, formerly of Cornell Paperboard Products Co., is now Division Plant Manager of Driscoll's Standard Coatings, Chicago, Ill.

Stanislaw Dominik, formerly of the Irving Pulp and Paper, Ltd., is now Chemical Engineer with the Distillers Corp., Ltd., Ville La Salle, Que.

Anthony L. Ensink is now Consultant on Graphic Arts Research for A. B. Dick Co., Chicago, Ill.

D. C. Everest is now Chairman of the Board, Marathon Corp., Rothschild, Wis.

Carl Gustav Geiger is now Technical Assistant to the President of Fiskeby Fabriks A/B, Norrkoping, Sweden.

Kenneth M. Grasse, formerly of the Economic Cooperation Administration, is now Research Chemical Engineer, Aetna Paper Div., Howard Paper Mills, Inc., Dayton, Ohio.

Howard C. Graham, formerly of the Crown Zellerbach Corp., is now Technical Supervisor, Elk Falls Co., Ltd., Campbell River, B. C.

F. D. Helverson is now Chief Industrial Engineer, Crown Zellerbach Corp., San Francisco, Calif.

Morris Housen is now Chairman of the Board, Erving Paper Mills, Erving, Mass.

Charles S. Huestis is now Manager of Container Board Manufacture of Robert Gair Co., Inc., New York, N. Y , and Vice-President and Assistant to the President for Southern Paperboard Co., Port Wentworth, Ga.

Lester M. Kapp, formerly of Pensacola, Fla., is now Plant Electrical Superintendent for the St. Regis Paper Co., Jacksonville, Fla.

Robert J. Kindl, Assistant Project Engineer for the Rust Engineering Co., has transferred from Pittsburgh, Pa., to Birmingham, Ala.

Robert Kinne, who has completed 16 months on a tour of duty with the Naval Reserve, has returned to the National Paper Corp., Ransom, Pa., as Night Superintendent.

Earl O. Knapp, formerly of the Weston Paper & Mfg. Co., is now Director of the Quality Control Laboratory of the Hinde & Dauch Paper Co., Sandusky, Ohio.

Daniel V. Lent, formerly Instructor in the Pulp & Paper Dept., College of Forestry, State University of New York, is now Chemist for W. R. Grace & Co., New York, N. Y.

John R. Lindquist, formerly of Certain-Teed Products Corp., is now Technical Representative for Stein, Hall & Co., Chicago, Ill.

O. B. Mason is now Division Manager of the Mead Corp., Chillicothe, Ohio.

Robinson E. Matthews is now Sales Manager of the Ecusta Paper Co., Pisgah Forest, N. C.

J. H. McCarthy is now Chief Engineer, Pulp and Paper Div. of St. Regis Paper Co., Jacksonville, Fla.

T. E. Powers, Technical Service Engineer for Hercules Powder Co., has transferred from Milwaukee, Wis., to Atlanta, Ga.

A. M. Ream is now Electrochemical Superintendent and Technical Assistant for Pulp for the Ecusta Paper Co., Pisgah Forest, N. C.

Joseph Rossman has moved his law office from Jefferson Place to Room 629, Du Pont Circle Building, Washington 6, D. C.

H. A. Rothschild is now Assistant to the Manufacturing Vice-President of the Kimberly-Clark Corp., Neenah, Wis.

E. Schoenberg is now in charge of the Straw Research Station, Algemeene Kunstzijde Unit, N. V., Arnheim, Holland.

M. O. Schur is now Research and Development Manager, Ecusta Paper Co., Pisgah Forest, N. C.

George E. Scofield is now Assistant Resident Manager, Rayonier, Inc., Fernandina, Fla.

George H. Sheets is now Assistant to the Division Manager, The Mead Corp., Chillicothe, Ohio.

John C. Stewart, formerly of Goslin-Birmingham Mfg. Co., is now Sales Engineer for Clark & Vicario, Bronxville, N. Y.

U. J. Westbrook is now Manager of Production of the Southern Division of St. Regis Paper Co., Pensacola, Fla.

William F. Woodzicka, formerly of the Rhinelander Paper Co., is now Mechanical Engineer for the U. S. Bureau of Reclamation, Columbia Falls, Mont.

* * *

The office of Rayonier, Inc., has been moved from 122 E. 42nd St. to 161 E. 42nd St., New York 17, N. Y. *W. E. Breitenbach*, *R. F. Erickson*, and *R. M. Pickens* will be located at the new address.

* * *

The New York Office of the Riegel Paper Corp. has been moved from 342 Madison Ave. to 260 Madison Ave.

* * *

The headquarters of the United Board & Carton Co. has been transferred from Syracuse, N. Y., to 2 Park Ave., New York 16, N. Y. *W. E. Stuhr*, President, is the official corporate representative in TAPPI.

* * *

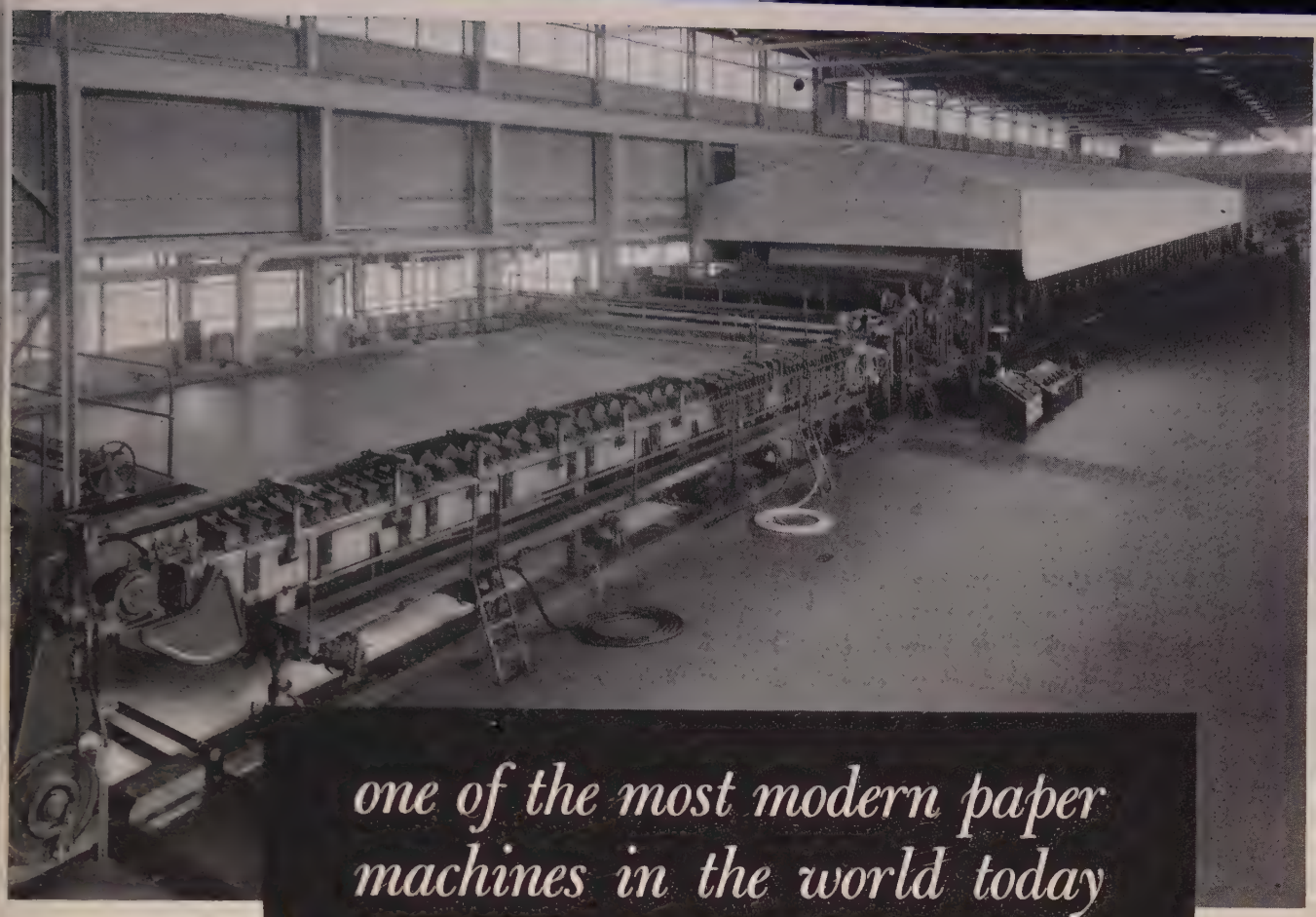
Charles D. McDuffie, President of William L. Barrell Co., 52 Chauncy St., Boston, Mass., has succeeded *Robert L. Tonner* as official company representative in TAPPI.

* * *

Louis B. Taylor has succeeded *R. W. Miller* as official representative of the Columbia-Southern Chemical Corp., Fifth Ave. at Bellefield, Pittsburgh, Pa.

NEW TAPPI CORPORATE MEMBERS

Alton Boxboard Co., Alton, Ill.



one of the most modern paper machines in the world today

We have designed and built what we believe to be one of the most modern paper machines in the world today. As the modern paper machine has grown in width and speed, it has outgrown mechanical controls . . . but we believe hydraulics is an ideal means of control. This Bagley & Sewall machine is the most completely hydraulic-controlled paper machine in the world. Each major operating unit requiring movement—each pressure operation and control is at finger-tip selection at a station point or console . . . providing instantaneous and accurate operation of the entire machine for the utmost control and productivity.

Recently installed at the St. Regis Paper Company, Cantonment, Florida, this machine has a wire width of 228" and was designed for a production of 400 tons per day at a speed of 2200 feet per minute.

designers and builders of paper making machinery since 1853

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WATERTOWN NEW YORK

Finland Representative
Aktiebolaget Ekstroms Maskinaffar
Helsingfors, Finland

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Cartonnerie de Kayserberg S. A. Kayserberg-Alspach-Haut-Rhine, France
Colthrup Mills, Thatchem, Berks, England
Columbia Cellulose Co., Ltd., Prince Rupert, B. C., Canada
Courtaulds (Ala.), Inc., P.O. Box 1076, Mobile, Ala.
De La Roza, Corp., New York, N. Y.
Drinmagh Paper Mills, Ltd., Dublin, Eire
Great Northern Paper Co., Bangor, Me.
Hudson Pulp & Paper Co., New York, N. Y.
Junckers Savvaerk A/S, Koge, Norway
Laboratoire Central des Services Chimiques de L'Etat, Paris, France

New TAPPI Sustaining Members

Albany Felt Co., Albany, N. Y.
Biggs Boiler Works Co., Akron, Ohio
Crosby Chemicals, Inc., De Ridder, La.
Earl Paint Co., Utica, N. Y.
Fabri-Valve Co. of America, Portland, Ore.
Graton & Knight Co., Worcester, Mass.
Howard Publishing Co., Chicago, Ill.
Industrial Nucleonics Corp., Columbus, Ohio
Keratene Co., Inc., Winsted, Conn.
Lauhoff Grain Co., Danville, Ill.
Manchester Machine Co., Middletown, Ohio
Nopco Chemicals Co., Harrison, N. J.
Roots-Connersville Blower Division of Dresser Industries, Inc., Connersville, Ind.
Rotareaed Corp., Bronxville, N. Y.
S. & S. Corrugated Machinery Co., Inc., Brooklyn, N. Y.
Trundle Associates, Cleveland, Ohio
E. C. Wolferz Alloy Equipment, Belleville, N. J.

Industry Notes

Freight

According to data released by the ICC, the pulp and paper industry used 1,672,104 cars for their raw materials and finished products in 1951. The tonnage involved amounted to 55,421,745 tons and railroad revenue derived, $356,838,338. Commodities shipped were pulpwood, wood pulp, newsprint, printing paper, wrapping paper, paper bags, paper and paper articles, and paperboard.

Statistics Conference

TAPPI and the University of Maine will conduct a conference on the application of statistical methods to the problems of the pulp and paper industry at the University of Maine, Aug. 4–16, 1952. A few more individuals can be accommodated. Details are available from Prof. Spoffard H. Kimball, University of Maine, Orono, Me.

Taxes

Taxes paid by the paper and paperboard industry increased by $27.60 a ton from 1949 to 1951. These costs will be even higher in 1952.

Big 6

The pulp and paper industry is the sixth largest industry in the United States. It employs upward of 254,000 workers; has an annual production in excess of 7.5 billion dollars; a capital investment of more than 6 billion dollars; annual wages to the workers in the industry exceeding 889 million dollars; pays total annual taxes upward of $1,102,000,000; and provides tools for workers with the investment estimated at an average of $27,000 per worker.

Planned New Capacity

Below is the tabulation of a revised Survey of New Capacity for 1951 through 1955 for the paper and board industry, and a summary of new capacity for 1951 through 1955 for wood pulp (prepared by the American Paper and Pulp Association). Data for 1951 through 1955 for the paper and board industry represent a revision of previously published capacity, due to the fact that installations of new machinery and improvements of existing machinery may not occur when scheduled. All annual papermaking capacity listed herein is on the basis of a 310-day year. Wood pulp capacity as included in this report is on the basis of normal full-time operations of the individual reporting mill.

This report may be summarized as follows:

The detail of planned capacity increases in the various grades of paper and kinds of wood pulp will be found on the following pages. Data on wood pulp capacity are from the United States Pulp Producers Association, Inc. Through the cooperation of the Writing Paper Manufacturers Association and the National Paperboard Association, additional data on new papermaking machinery not reported to this Association have been included, so that the capacity for new machine installations is believed to be complete for the paper and board industry.

This report is a revision of our report dated Oct. 2, 1951, and constitutes principally a revision of the new machine data for paper and board. To the data for rebuilt machines included in our October 2 report have been added some data received from the National Paperboard Association.

The machine start-ups for 1953 are not too certain in many instances. Our observation of past behavior in similar circumstances indicates that it is characteristic of this type of survey that data for more than 2 yr. in the future are not altogether reliable. The reasons for this are mainly (1) uncertainty and delays on the part of machine builders and (2) changes in plans by management.

Our report includes only projects for which orders have been placed for paper machines and other projects for increasing capacity for which we believe firm commitments have been made.

We are aware that Certificates of Necessity have been issued by the DPA for some projects not included in this report. These may result in additional or somewhat altered increases in capacity. But the present report has not taken these into account as being too nebulous at the present time.

As a consequence of this situation we have headed the final column as 1953–55.

On the above basis capacity increases for the years, 1951, 1952, and 1953–55 by grades of paper and board are shown below, in tons, in per cent of total increase, and by percentages indicating the distribution of the capacity increases during the period:

	Total wood pulp, tons	*Total paper and board, tons*	*Paper, tons*	*Board, tons*
Estimated capacity				
End of 1950	17,123,889	24,480,820	12,562,620	11,918,200
End of 1951	18,406,957	25,346,495	13,050,715	12,295,780
End of 1952	19,692,536	26,532,865	13,669,785	12,863,080
End of 1955	....	27,750,855	14,083,325	13,667,530
Percentage increase				
1951/1950	+7.5	+3.5	+3.9	+3.2
1952/1950	+15.0	+8.4	+8.8	+7.9
1953–1955/1950	....	+13.4	+12.1	+14.7

A WATER SUPPLY THAT'S *ALWAYS* READY WHEN YOU NEED IT

The 125,000-gal. Horton elevated water tank at the Dunn Paper Company is 100 ft. to bottom. A portion of its capacity is used to supply process water and the remainder is held in reserve at all times for fire protection.

The Dunn Paper Company—manufacturers of machine-glazed bleached, semi-bleached, and unbleached sulphite and kraft specialties—recently installed the Horton elevated tank shown on the right at their Port Huron, Michigan plant. The 125,000-gal. steel structure supplies water for fire protection and also for various paper making processes.

Like many mills, Dunn Paper Company installed an *elevated* tank to provide a *dependable* supply of water whenever it is needed and to guard against interruptions in production. These are proven advantages because an elevated water supply is backed by gravity pressure. Service is assured 24 hours a day!

Horton elevated water tanks with ellipsoidal-bottoms are built in standard capacities from 15,000 to 500,000 gallons and Horton Waterspheres in sizes from 25,000 to 250,000 gallons. The welded steel construction of Horton tanks provides smoother surfaces for easier painting. Maintenance is reduced to a minimum.

Horton tanks providing gravity pressure for automatic sprinkler systems are built to meet the requirements of either the National Board of Fire Underwriters, the Inspection Department of the Associated Factory Mutual Fire Insurance Companies or the Factory Insurance Association, as required.

For more information or quotations on Horton elevated tanks and their profitable application in your plant for either general service or fire protection—write our nearest office without obligation. Please state type of service desired, height to bottom, capacity, location where tank is to be erected, and type of insurance carried.

CHICAGO BRIDGE & IRON COMPANY

Atlanta, 3 . . . . . 2142 Healey Building
Birmingham, 1 . . . . . 1559 North 50th Street
Boston, 10 . . . . . 1064—201 Devonshire Street
Chicago, 4 . . . . . 2130 McCormick Building
Cleveland, 15 . . . . . 2255 Guildhall Building
Detroit, 26 . . . . . 1565 Lafayette Building
Havana . . . . . 402 Abreu Building
Houston, 2 . . . . . 2163 National Standard Building
Los Angeles, 17 . . . . 1561 General Petroleum Building
New York, 6 . . . . . 3333—165 Broadway Building
Philadelphia, 3 . . . 1665—1700 Walnut Street Building
San Francisco, 4 . . . . . 1577—200 Bush Street
Seattle, 1 . . . . . 1368 Henry Building
Tulsa, 3 . . . . . 1666 Hunt Building
Washington 6, D. C. . . . . 1134 Cafritz Building

Plants in BIRMINGHAM, CHICAGO, SALT LAKE CITY, and GREENVILLE, PA. In Canada: HORTON STEEL WORKS, Limited, FORT ERIE, ONT.

Grade of paper	1951–55 capacity increase to total, %	1951–55 capacity increase, tons	Per cent of total 1951–55 increase during 1951	1952	1953–55
Fine paper	2.0	66,650	6.5	88.8	4.7
Book and groundwood papers	3.1	101,060	75.5	15.3	9.2
Tissue paper	3.3	106,950	36.8	50.1	13.1
Wrapping and specialties	29.9	978,050	35.6	47.0	17.4
Newsprint	8.2	267,220	7.3	11.6	81.1
Miscellaneous	...	775	100.0		
Total paper	46.5	1,520,705	32.1	40.7	27.2
Paperboard, including wet machine and building board	53.5	1,749,330	21.6	32.4	46.0
Total paper and board	100.0	3,270,035	26.5	36.3	37.2

Capacity Increases—New and Rebuilt Machines (Tons)

Grade of paper	Capacity at end of 1950	Increases during 1951	Capacity at and of 1951	Increases during 1952	Capacity at end of 1952	Increases during 1953–55	Capacity at end of 1955
Fine paper	1,285,030	4,340	1,289,370	59,210	1,348,580	3,100	1,351,680
Book and groundwood papers	3,407,220	76,260	3,483,480	15,500	3,498,980	9,300	3,508,280
Tissue papers	1,369,740	39,370	1,409,110	53,630	1,462,740	13,950	1,476,690
Wrapping and specialties	3,967,580	347,820	4,315,400	459,730	4,775,130	170,500	4,945,630
Newsprint	1,032,810	19,530	1,052,340	31,000	1,083,340	216,690	1,300,030
Miscellaneous	111,240	775	112,015	.....	112,015	.....	112,015
Building paper	1,389,000	...	1,389,000	.....	1,389,000	.....	1,389,000
Total paper	12,562,620	488,095	13,050,715	619,070	13,669,785	413,540	14,083,325
Paperboard, including wet machine and building board	11,918,200	377,580	12,295,780	567,300	12,863,080	804,450	13,667,530
Total paper and board	24,480,820	865,675	25,346,495	1,186,370	26,532,865	1,217,990	27,750,855

Capacity Increases

New Machines (Tons)

Grade of paper	1951 Daily	1951 Annual basis	1952 Daily	1952 Annual basis	1953–55 Daily	1953–55 Annual basis
Fine paper	30	9,300	181	56,110	..	.....
Book and groundwood papers	60	18,600	..	...	..	.....
Tissue paper	90	27,900	93	28,830	..	.....
Wrapping and specialties	822	254,820	1405	435,550	500	155,000
Newsprint	..	...	100	31,000	660	204,600
Miscellaneous	..	...	..	...	..	.....
Building paper	..	...	..	...	..	.....
Total paper	1002	310,620	1779	551,490	1160	359,600
Paperboard	309	95,790	690	213,900	2175	674,250
Building board	..	...	400	124,000	..	.....
Total paper and board	1311	406,410	2869	889,390	3335	1,033,850

Rebuilt Machines (Tons)

Fine paper	−16	−4,960	10	3,100	10	3,100
Book and groundwood papers	186	57,660	50	15,500	30	9,300
Tissue paper	37	11,470	80	24,800	45	13,950
Wrapping and specialties	300	93,000	78	24,180	50	15,500
Newsprint	63	19,530	..	...	39	12,090
Miscellaneous	2½	775	..	...	..	.....
Building paper	..	...	..	...	..	.....
Total paper	572½	177,475	218	67,580	174	53,940
Paperboard	909	281,790	740	229,400	420	130,200
Building board	..	...	..	...	..	.....
Total paper and board	1481½	459,265	958	296,980	594	184,140

Wood Pulp Capacity by Grades, 1951–55

January 1	Total wood pulp	Dissolving and special alpha	Paper grades sulphite	Sulphite bleaching capacity	Paper grades sulphate
1951	17,123,889	554,940	2,578,344	1,904,214	8,337,287
1952	18,406,957	715,346	2,587,326	1,928,674	9,144,157
1953	19,692,536	857,096	2,607,505	1,942,957	9,940,564
1954	20,874,169	1,141,421	2,607,505	1,942,957	10,654,319
1955	21,016,169	1,240,421	2,607,505	1,942,957	10,697,319

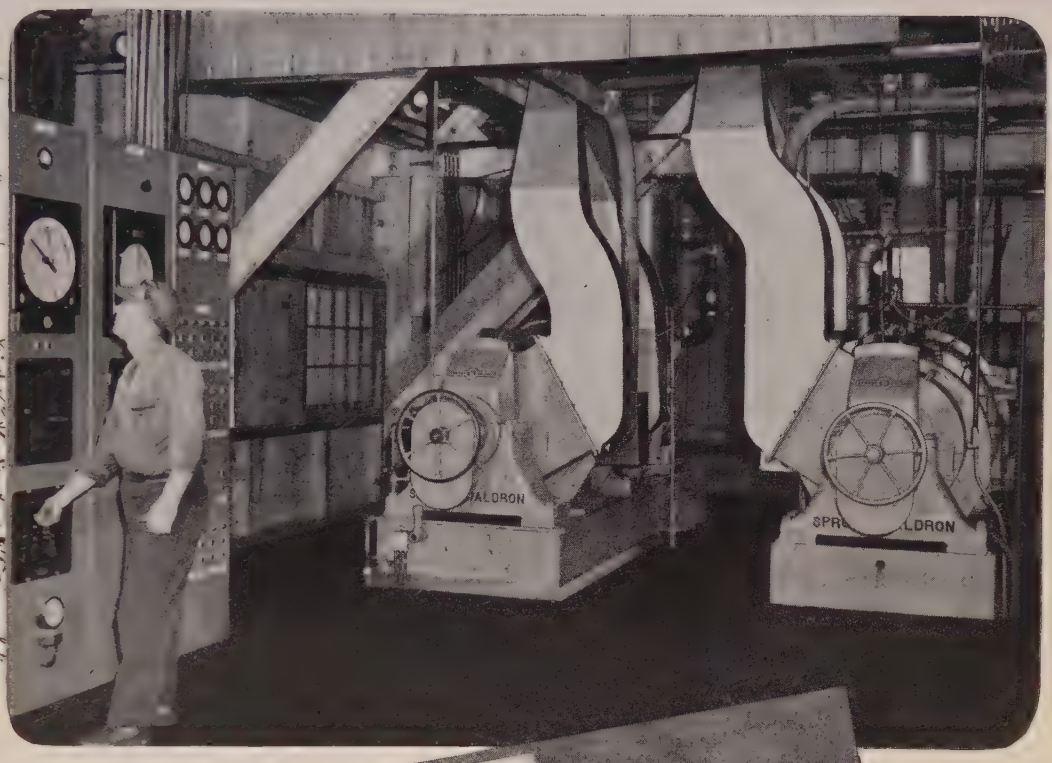
January 1	Sulphate bleaching capacity	Soda	Groundwood	Groundwood bleaching capacity	All other grades
1951	2,295,871	466,937	2,908,225	424,299	2,278,156
1952	2,613,228	482,917	2,923,236	618,246	2,553,975
1953	2,777,388	534,817	2,945,243	619,906	2,807,311
1954	2,942,013	534,817	2,976,246	619,906	2,959,861
1955	2,951,013	534,817	2,976,246	619,906	2,959,861

Hardboard To Solve Pulping Problems ECONOMICALLY

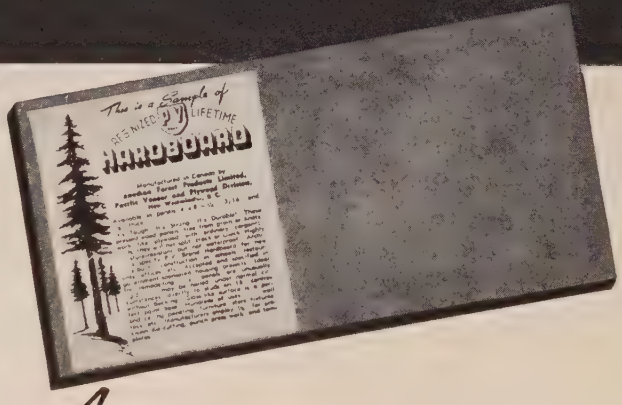
Canadian Forest Products, L't'd.

PACIFIC VENEER AND PLYWOOD DIV.

Chooses SPROUT-WALDRON'S



Two Sprout-Waldron 36-2 Refiners prepare pulp from Asplund Fiber for the well-known P. V. BRAND hardboard.



- High pulp quality
- High capacity
- Flexibility of operation
- Rugged construction
- Low maintenance

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This Sprout-Waldron technical publication reports important information on new and improved pulping processes—including semichemical pulping.

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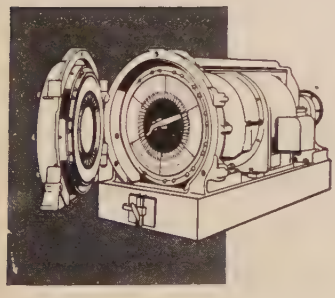
S W



SPROUT-WALDRON

PULP REFINERS

230

NOTES REGARDING WOOD PULP CAPACITY DATA

1. Tabulated data subsequent to 1951 are preliminary and subject to revision as new programs are initiated, as indefinite programs become definite, or as additional information is developed.
2. Tabulated data include only reasonably assured capacity increases resulting from improvements, speed-ups, or new plants. Capacity data for the following new mills, scheduled for completion after 1/1/52, are included in the tabulation:

Buckeye Cotton Oil Co.
Ketchikan Pulp Co.
Olin Industries, Inc.
Rome Kraft
St. Joe Paper Co.
Weyerhaeuser Timber Co., Longview and Everett, Wash.
International Paper Co., Natchez, Miss.
National Container Corp., Valdosta, Ga.
Rayonier, Inc., Ga.
Tuberoid Co., Dallas, Tex.
St. Regis Paper Co.

3. Not included in the tabulation are:
   (a) A number of projects definitely planned, but indefinite as to date of completion; new capacity involved in such projects, on an annual basis amounts to approximately 500,000 tons.
   (b) A number of proposed new mills, publicly announced, but in respect to which there is as yet no definite assurance of completion; new capacity involved in such projects, on an annual basis, amounts to 850,000 tons. (Plans for some of this expansion have already been abandoned for the present and there may be further attrition in this category.)
4. The current expansion program, therefore, may be summarized as follows:

18,407,000 tons—Total capacity as of Jan. 1, 1952
2,609,000 tons—Scheduled for completion by 1955
500,000 tons—Probable, but not yet scheduled
850,000 tons—Possible, but not yet definite
22,366,000 tons—Potential annual capacity by early 1955 if all announced projects are completed

DEFINITIONS

Annual capacity is the tonnage that could be produced on an annual basis with full use of equipment and adequate supply of pulpwood, chemicals, and labor. The annual capacity of each mill is its reported daily capacity multiplied by the number of days per year that it expects, under normal conditions, to operate, and not necessarily its present operating schedule. New capacity installed in a given year is included only for the number of days that it will be operative in that calendar year. All data are in tons of 2000 lb., air-dry weight.

SOURCE AND COVERAGE

Companies whose aggregate annual capacity, as of Jan. 1, 1952, amounted to 96% of total industry capacity have supplied the United States Pulp Producers Association, Inc. with information in respect to their current capacities and planned changes in capacity. In compiling industry totals, it has been assumed that the capacity of those companies that have not reported will continue at the Jan. 1, 1952, level.

Analysis of Certificates

It is difficult, if not impossible, to balance the volume of Certificate of Necessity increases against the planned new capacity as shown by the American Paper and Pulp Association survey. In the case of at least several certificates that have been issued there is considerable uncertainty as to whether they will result in actual capacity increases. In the present revised American Paper and Pulp Association survey are listed only projects for which paper machines have been completed or ordered, or for which there is a commitment to install improvements. Comparing these factual data with the somewhat more uncertain Certificate of Necessity data, the following balances are developed from the DPA Expansion Program announced March 20, 1952, for these certificates. The plus sign indicates an excess in capacity of the American Paper and Pulp Association survey over that of the DPA Expansion Program. The period covered is from Jan. 1, 1951, to Jan. 1, 1956.

Coarse papers	+498,000 tons
Book and groundwood papers	−124,000 tons
Fine paper	−33,000 tons
Tissue paper	−67,000 tons
Newsprint	−227,000 tons
Building paper	−10,000 tons
Paperboard	+731,000 tons

It will be seen from the above table that present planned new capacity falls short of the Certificates of Necessity that have been issued in the printing grades, newsprint, book and groundwood, and fine paper, by a total of 384,000 tons annual production. Building papers are apparently short by 10,000 tons, and tissue paper by 67,000 tons annual capacity. On the other hand, coarse paper capacity and paperboard capacity will be in excess by 1,229,000 tons annual capacity at the end of the period.

An analysis of these latter items indicates that some Certificates of Necessity may have been issued in excess of the announced DPA Expansion Program. However that may be, it seems clear that planned new capacity in coarse papers and paperboard exceeds the DPA Expansion Program, while the prospective increases expected by the DPA in the other grades are not yet programmed by industry.

Certified Pulp Testers

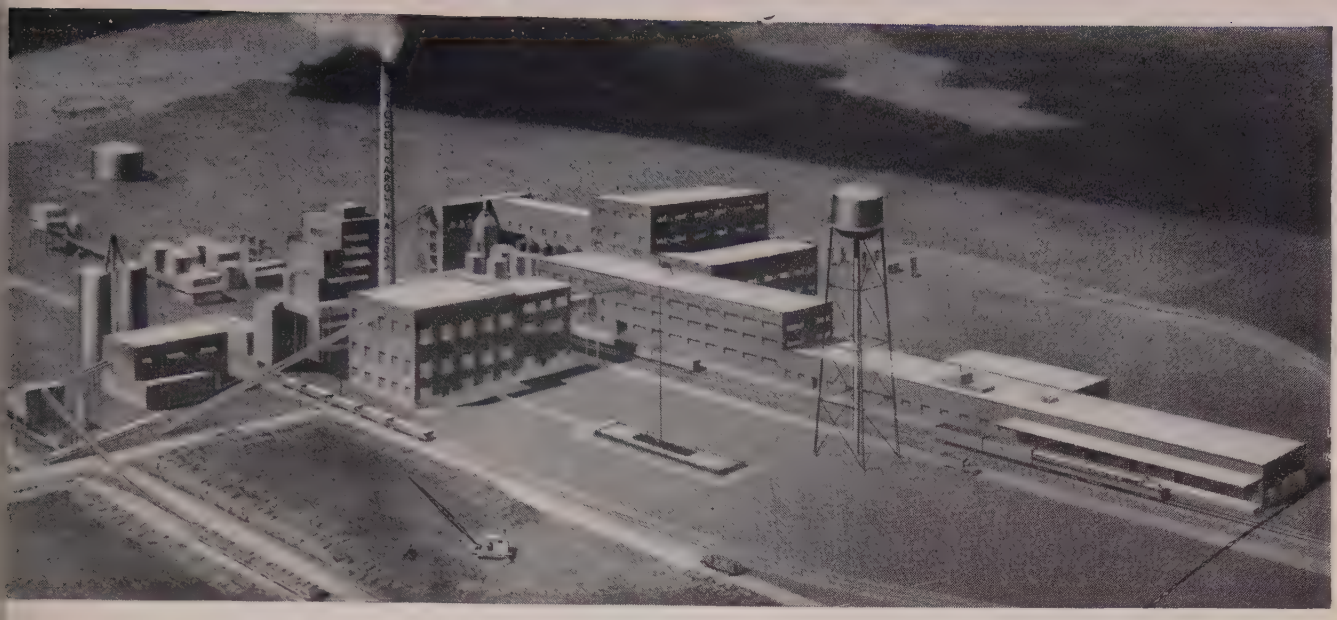
The Certified Pulp Testers Bureau (TAPPI, APPA, and AAWPI) has issued an up-to-date list of the individuals and laboratories that have been certified to test wood pulp for weight and moisture content on a commercial basis as follows:

Baker, D. H., H. R. MacMillan Export Co., Ltd., Pulp Div., P.O. Drawer 18, Nanaimo, B. C., Canada
Bradford, Harry C., Rex Paper Co., Kalamazoo, Mich.
Bringman, Robert E., St. Joe Paper Co., Port St. Joe, Fla.
Brown, W. W., Howe Sound Pulp Co. Ltd., Port Mellon, B. C., Canada
Butler, R. A., Pacific Mills, Ltd., Ocean Falls, B. C., Canada
Clark, Clarence T., Restigouche Co., Ltd., Campbellton, N. B., Canada
Enghouse, Clarence A., Crown Zellerbach Corp., West Linn, Ore.
Hartnagel, Nelson, Fiberboard Products, Inc., Port Angeles, Wash.
Ingram, C. D., Hollingsworth and Whitney Co., P.O. Box 1189, Mobile 7, Ala.
Jacoby, W. C., Crown Zellerbach Corp., Camas, Wash.
McLean, E. A. G., MacMillan & Bloedell (Alberni) Ltd., Port Alberni, B. C., Canada
Marriott, Robert R., Crown Zellerbach Corp., Port Townsend, Wash.
Moden, F. P., Anglo-Canadian Pulp and Paper Mills, Ltd., Que., P. Q., Canada
Sherk, Dr. D. L., Columbia Cellulose Co. Ltd., Engineering Dept., P.O. Box 1000, Prince Rupert, B. C., Canada
Smith, C. L., National Container Corp., Jacksonville, Fla.
Tennent, G. R., Hummel-Ross Div., Continental Can Co., Hopewell, Va.
Thieme, R. I., Soundview Div., Scott Paper Co., Everett, Wash.
Weleber, F. J., Publishers Paper Co., Oregon City, Ore.
Andrews, A. B., Box 57, Lewiston, Me.
Danneker & Evans, Inc., 309 Camp St., New Orleans, La.
Donald & Co., J. T., Ltd., 1181 Guy St., Montreal, Canada
Goodrich, Neil C., South Haven Terminal Co., Inc., 251 Center St., South Haven, Mich.
Gulick-Henderson Laboratories, 431 S. Dearborn St., Chicago 5, Ill.
Hamlin & Morrison, 216 S. 2nd St., Philadelphia, Pa.
Landes, William, Paper and Pulp Testing Laboratory, 118 E. 28th St., New York, N. Y.
Orthmann Laboratories, Inc., 922 N. 4th St., Milwaukee, Wis.
Skinner & Sherman, Inc., 246 Stuart St., Boston, Mass.
Sutliff, John H., Font Grove Rd., Slingerlands, N. Y.
U. S. Testing Co., Inc., 1415 Park Ave., Hoboken, N. J.
Whittington, E. F., 530 W. Cedar St., Kalamazoo 45, Mich.
Wiley & Co., Inc., 904 N. Calvert St., Baltimore, Md.
Williams Apparatus Co., Inc., Herald Bldg., Watertown, N. Y.

West Virginia

The manufacture of precipitated calcium carbonate, produced by the Industrial Chemical Sales Division of West Virginia Pulp and Paper Co. in a plant located at the company's mill in Covington, Va., will be discontinued October 31, it was announced recently by company officials.

Precipitated calcium carbonate is a fine white powder, made from lime, which is used widely in the manufacture of drugs and pharmaceuticals and employed in a number of



71 Ingersoll-Rand Pumps installed at

Riegel-Carolina's New Pulp Mill

Ranging in size from ¾ to 400 hp, they serve throughout plant

Riegel-Carolina Corporation's new 20 million dollar bleached kraft specialty pulp mill at Acme, N. C. is being rushed to completion and is scheduled for full-scale operation early this year. The 45-acre site will produce 200 tons of bleached sulphate pulp daily from pulpwood grown on the company's holdings in the nearby Waccamaw forest. Modern in every detail, this new mill takes advantage of all the recent economies developed in the production of bleached kraft pulp.

Ingersoll-Rand pumps serve in practically every phase of the pulp producing process, handling stock of varying consistencies, black liquor, bleach chemicals and general service water. They range in size from Motorpumps of ¾ horsepower to 400 horsepower boiler feed units. The latter are ultra-modern Class HMTA 6-stage pumps, designed with a Unit-Type rotor assembly for sustained high efficiency and easy maintenance.

Modern pulp and paper mills can find the best solution to their varied pumping problems in Ingersoll-Rand's complete pump line. An experienced field engineer will be glad to study your problems and make his recommendations for efficient, dependable pumps to suit your particular process.

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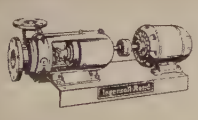
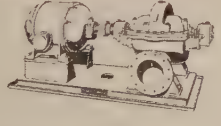
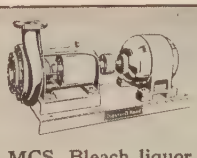
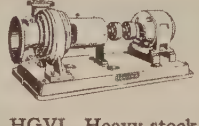
Here are a few of the pumps installed and their services:


CORVL, Black liquor


AFV, White water

HMTA, Boiler feed


MCS, Bleach liquor


HGVL, Heavy stock

important industrial processes. The Covington product, known locally as "chalk," has been marketed by the Industrial Chemical Sales Division under the familiar brand name of Snow Top. Additional tonnages of "wet chalk" made at the Covington plant have been used as a filler for certain grades of printing paper. Both operations will be discontinued.

Crawley Williams, Manager of the Covington mill, said that rising manufacturing costs, combined with a definite market trend from the highly controlled production of quality grades to heavy volume production for industrial users, prompted the decision to close down the chalk operation.

"For years the bulk of our precipitated calcium carbonate production was devoted to grades meeting exacting purity and uniformity standards, for use in the manufacture of such products as antibiotics, tablets of all kinds, dentifrices, chewing gum and confections, food and the like," Mr. Williams explained.

"We were able to make 'chalk' profitably for these markets because the emphasis was on the quality of the product manufactured at Covington," he continued. "In recent years, however, other chemical products have invaded the drug and pharmaceutical fields. The chief market for chalk is now for technical applications in industry where, because of the great volumes used, price is the all-important consideration.

"With plant facilities geared to quality operations, and high manufacturing costs, we cannot compete in this mass market without costly and extensive changes in equipment. The prices available in the mass market do not promise sufficient return to justify the capital outlay required. This leaves us with no practical alternative but to shut down the chalk plant. None of our other operations will be affected."

Mr. Williams said the chalk plant would continue production for the next five months in order to fill existing orders and give customers an opportunity to establish other sources of supply. The shutdown will take place at the end of the company's fiscal year.

The company first began manufacturing precipitated calcium carbonate in 1912 at its Luke, Md., plant, where the electrolytic plant had an excess of lime capacity and CO_2 gas, required in the process.

Later the company perfected a process for using "wet chalk" as a filler for paper and additional chalk plants were established at the Covington and Tyrone, Pa., mills.

At the Covington plant, which began operations in 1928, an improved process was developed which gave the company a superior brand of the dry—or "precipitated"—product. After this process was perfected, the manufacture of all calcium carbonate for outside sale was transferred to Covington. For many years the output of the plant, though subject to the normal peaks and valleys of the chemical industry, met with a strong demand. The market swing to the technical grades began to be felt more than a year ago, when the plant work schedule was reduced from a seven-day to a five-day week, marked by occasional shutdowns.

While the "chalk" operation has been a long and successful one, due to the development and marketing skills of the Industrial Chemical Sales Division and the quality standards adhered to by the operating personnel, the volume of the business is small in relation to that of the mill as a whole, it was pointed out.

Mr. Williams said the shutdown of the chalk plant will eliminate about 55 jobs. So many factors are involved, he pointed out, that it is impossible to determine this far in advance what net reduction in the mill's over-all working force, which now numbers around 2300, may be required. In any event, it was stated, the seniority rights of those affected will be worked out, according to the contract, by joint discussion between the company and the union.

Vanillin

Givandan-Delawanna who has long produced vanillin from engenol, expects to start commercial production of vanillin from lignin this month.

Otto Maas

Otto Maas, Professor of Chemistry at McGill University, was awarded the medal of the Chemical Institute of Canada at Montreal in June. His war work was on explosives and the organization of laboratories to serve the national need.

Southeast Drainage Basins

The Federal Security Agency, Public Health Service, Washington, D. C., has issued the 13th volume of its Water Pollution Series on "Southeast Drainage Basins."

Calibrating Foil

For some years, the Hurlbut Paper Co. has been providing a service to paper mills and others in the paper industry in supplying them, at a nominal cost, aluminum foil for the purpose of calibrating their Mullen testers.

While, in the beginning, there were not too many mills involved, the demand for this foil, both in the United States and abroad, has grown to a point whereby it has become a considerable burden to the company and it feels that it can no longer continue this service.

As soon as its present supply of foil has been exhausted, it will no longer be able to supply the foil to the trade. At the present writing, the company believes that it has on hand sufficient foil to take care of requirements for the next three or four months.

Machine Coating Patent

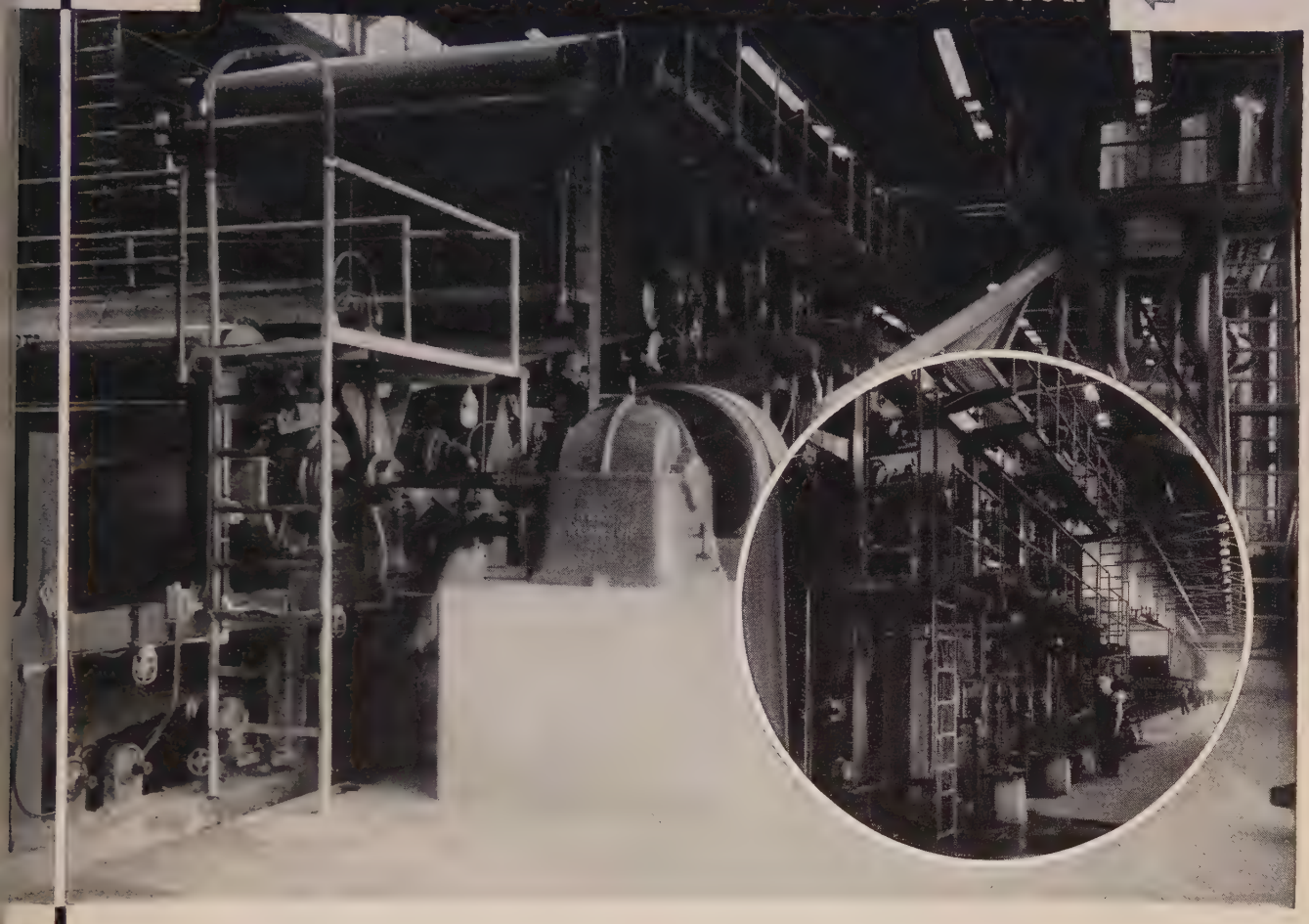
Milwaukee, Wis., June 2, 1952: In a memorandum decision in the important case involving the patent rights of the Consolidated Water Power & Paper Co. and Peter J. Massey versus Kimberly-Clark Corp., U. S. District Judge Robert E. Tehan, District Court, Eastern District of Wisconsin, on May 29 stated, "The Court is of the opinion that the defendant has not infringed plaintiffs' patent no. 1,921,368 and that plaintiffs' patent no. 1,921,368 is invalid."

The Memorandum Decision continues, "Civil Action No. 4373 having been commenced May 3, 1947, and Civil Action No. 4782 having been commenced July 21, 1949, and the two actions having been consolidated for trial by stipulation prior to trial and pursuant to order entered Dec. 7, 1949, and having come on for trial before the court without a jury on the 21st, 23rd, 24th, and 28th of February, 1950, and the 1st, 2nd, 3rd, 7th, 8th, and 9th of March, 1950, and the trial having been resumed for oral argument by counsel on the 19th day of October, 1950, and the court having carefully considered the testimony presented, the arguments of counsel, the briefs submitted, and the entire record in this matter, and being fully advised in the same" the Court feels that the patent has not been infringed and furthermore, that the patent is invalid.

The Memorandum Decision continues, "This Court will file its opinion in this matter at a later date. Within fifteen (15) days after receipt of the opinion which the Court shall file in this matter, the defendant shall submit proposed finding of fact and conclusions of law, after first serving a copy thereof upon the plaintiffs. Plaintiffs shall be allowed an additional fifteen (15) days to file objections, proposed changes or amendments."

Peter J. Massey on Aug. 8, 1933 received a patent, United States 1,921,368, "Process of Coating Paper." The claims of the Massey patent application as filed were rejected, but after an interview with the Examiner and having made amendments the patent was finally granted. However, patents obtained from the U. S. Patent Office are not necessarily valid

Latest Design and Remote Control Features Incorporated in the B-C Press Section



● Because remote control of equipment is the outstanding forward step in paper making since the war, Black-Clawson has gone "all the way."

A Black-Clawson press section with the primaries superimposed over the vat section available with remote control of practically every operation.

Loading and lifting of press rolls —by remote control. Motor operated oscillating doctors with broke conveyors. Push button operation of felt stretchers. Air operated clutches on press drives. Air operated felt guides.

Discuss this press—the entire machine in fact—from the standpoint of remote control operation with our representative on his next call.

THE BLACK-CLAWSON CO.

HAMILTON, OHIO

Divisions: SHARTLE BROS. MACHINE CO., Middletown, Ohio • *Western Sales Office:* Mayer Bldg., Portland, Oregon
DILTS MACHINE WORKS, Fulton, New York • *Southern Sales Office:* 937 Coventry Road, Decatur, Georgia
Associate: THE ALEXANDER FLECK LTD., Ottawa, Ont. • *Subsidiary:* B-C INTERNATIONAL LTD., Greener House, Haymarket, London, S. W. 1, Eng.

but are only presumptively valid. There are 29 different defenses which may be alleged, any one of which might upset the validity of the patent.

The present owners of the Massey patent, Consolidated Water Power & Paper Co. and Peter J. Massey, sued Kimberly Clark Corp. for the infringement of this patent. In their defense the defendants denied infringement and asserted that the Massey Process improvement was anticipated as shown in a number of prior-art references cited by the defendant and also asserted that if anticipation was not shown in these references, the Massey patent still lacked invention over the disclosure in these references.

The Patent Attorney for Kimberly-Clark Corp. asked: (1) Are the claims of the Massey patent broad enough to include defendant's operation of his machines? (2) If the claims are broad enough, are those claims valid when interpreted to include defendant's machines? (3) Are the plaintiff's, Consolidated and Massey, barred from court assistance because of unconscionable, i.e., unreasonable, delay in asserting their claim, during which time the defendant's relying thereon, expended large sums of money installing and adapting its paper machines to practice the accused process? (4) Are not the plaintiffs barred from maintaining this suit by entering certain license agreements which include the patent in suit and which contain provisions contrary to the Anti-Trust laws?

The defendants asserted that Massey tied all his claims to the idea of dehydration of the color coating by mechanical dehydration, before applying it to the paper. In short, all of Mr. Massey's claims required a "preworking" of the coating mixture in the transfer rolls before it gets to the applicator nip, in order to reduce the moisture content to a desirable minimum amount, so that the paper web will not be adversely affected, a process somewhat analogous to offset printing. Mr. Massey stressed his "preworking" of the coating material in order to overcome the Patent Office Examiner's rejection on a Thompson and also on a Wheelwright reference.

Furthermore, Kimberly-Clark Corp. asserted that a literal reading of the claims did not "read on" their process. They further asserted that Consolidated was bound by the limitation of "preworking" to effect dehydration, particularly since each claim was obtained because of this "preworking" differentiation over the prior art.

Consolidated Water Power & Paper Co. in the "file wrapper" and in the claims asserted that a "smooth" dehydrated coating film is transferred to the moving paper machine web, whereas Kimberly-Clark asserted that the film on their applicator roll was not smooth and therefore they did not transfer a "smooth" film as called for by Consolidated's patent claims.

In the petitions filed in connection with the suit, Consolidated Water Power & Paper Co. asserted that although Kimberly-Clark Corp. may not dehydrate by preworking, yet by using a dispersing agent they prepare a coating mixture of such high solids content that they do not need to prework the coating film. The brief for Consolidated Water Power & Paper Co. stated that their contribution was a process for coating paper on or between the sections of the paper machine, whereby no "pool" of water is formed at the applicator nip. In refutation of this the Kimberly-Clark brief asserted that this is not what the patent says or what the claims called for and furthermore that it is now too late to rewrite the patent; that the patent was never reissued in the corrected form, and that the patent expired on Aug. 8, 1950, and cannot be reissued.

In further substantiation to their claim the brief of the Consolidated Water Power & Paper Co. asserted the great financial success of their process, the fact other companies have taken out licenses under the patent, and that TAPPI gave Mr. Massey a gold medal in recognition of his contributions to the paper industry are proofs of the inventiveness claimed in the patent.

In rebuttal to this, the Kimberly-Clark brief stated that t financial success is not necessary proof of inventiveness but t may be due to good organization and salesmanship. Moreover, the fact that other companies accepted licenses under the patent is no proof of its validity and the fact that Mr. Massey received the TAPPI medal may be because of his over-all contributions to the paper industry and not because of the value of this particular patent.

Furthermore, the Kimberly-Clark Corp. asserted that they learned nothing from the patent, since relative to their process they obtained all the necessary know-how from the printed art prevalent prior to Mr. Massey's patent. Furthermore, the Kimberly-Clark Corp. brief asserted that the only thing the Massey patent teaches, if anything, is that an offset printing press may be used to apply coating.

The Kimberly-Clark Corp. also contended (E) that coating as a part of the papermaking operation, i.e., "on the paper-machine coating," or "machine coating," is not a patentable novelty to Mr. Massey because (1) the "reduced to practice on March 22, 1930" experiments were performed on an offset printing press that printed separate sheets, (2) the defendant's continuous method, viz., the G-K method, used between 1932 and the present on Defendant's Number 5 machine at Kimberly is not charged to infringe the patents in suit, (3) also the continuous process was old long prior to Massey; see Schumacher United States 726,358, Davidson and Gorges 890,221, and Wheelwright 1,195,888.

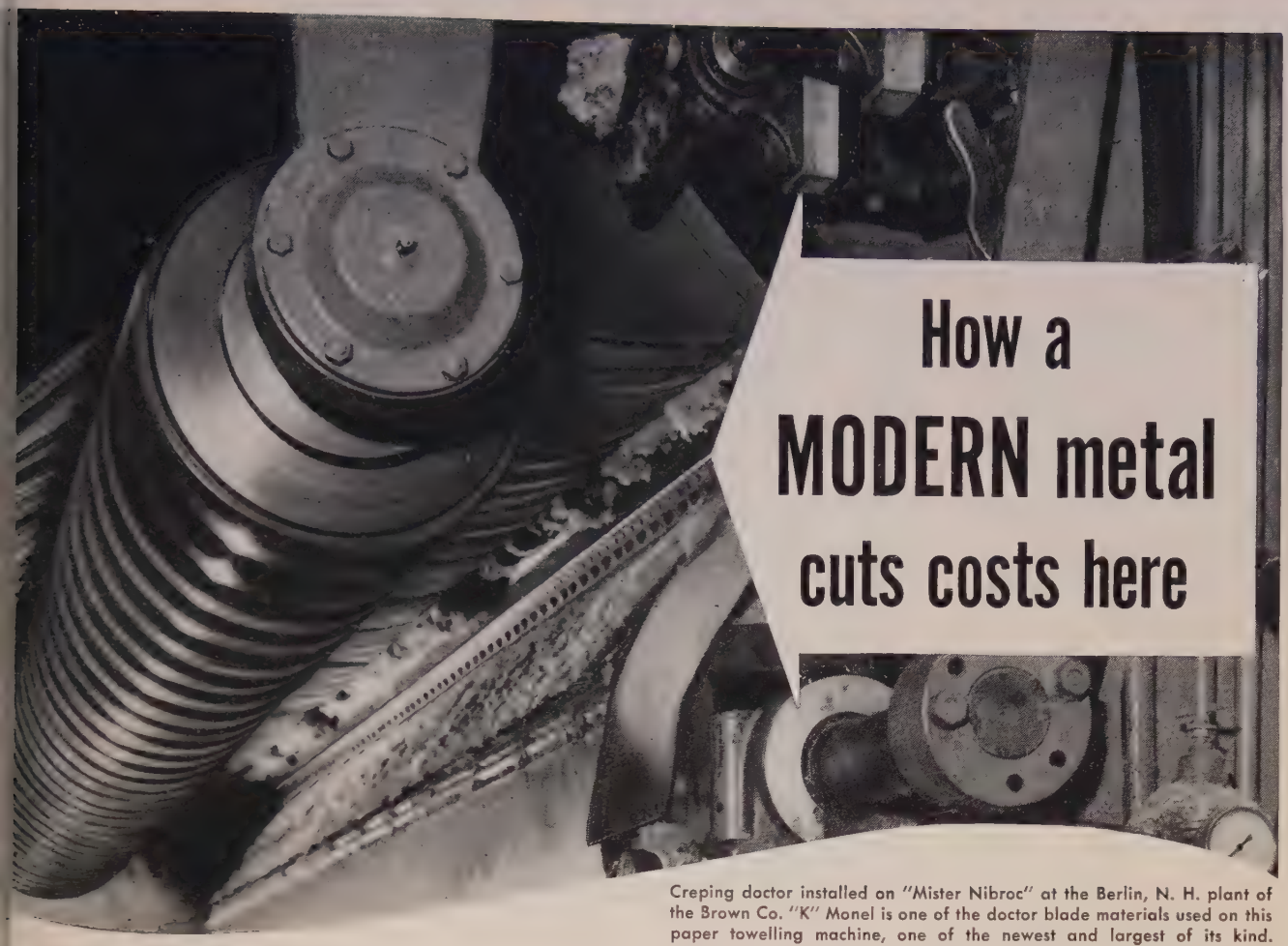
Kimberly-Clark also questioned whether there is patentable novelty in the process set forth in the patent. Mr. Massey stated in his patent that "suitable coating substances such as, for example, size, glue gelatin, casein, starch, clay calcium sulphate, resin oils and/or varnishes carried and/or dissolved in a vehicle and/or diluent of water or oil" may be used. Kimberly-Clark asserted this broad language includes inoperative embodiments and hence are misleading and objectionable, and they asserted that Consolidated cannot rewrite or restrict the patent to cover only clay-starch-water coating mixtures and furthermore, because of this broad language, which includes oils and varnishes, the prior art relating to oils and varnishes is applicable as references of rejection of the claims.

Moreover, the patent states that: "The proportion of liquid to soluble material may range in the approximate vicinity of 50%, more or less." Attention is directed to the fact that the amount of "liquid" is to be 50% of the "soluble material"; not solid material. It is difficult to understand what the "solids content" of the coating mixture may be. This ambiguous statement is probably due to poor patent draftsmanship or to not asking the inventor what the facts are, and thereafter setting them down.

Kimberly-Clark asserted that the feature of applying a coating without subsequently brushing or smoothing it is old as being inherent in a host of patents, viz. Wickel, Wheelwright, Davidson and Gorges, and Pohl and as being shown in Traquair, United States 1,518,371 who it is asserted shows that, "A sufficient quantity of water is absorbed by the paper before it passes from between the rolls that the coating is rendered immobile; that is, set or fixed so that it can no longer flow. The smooth surface formed by the rolls is therefore retained. . . ."

The defendants as a further defense alleged that Consolidated and Massey could not avail themselves of the Court assistance because they have violated the Anti-Trust laws in relation to unlawful monopolies in that one Seaman who owned the process patent on Dec. 2, 1933, licensed Consolidated to practice the processes and sell the products of the two Massey patents 1,921,368 and 1,921,369. Also the owners agreed among themselves to tie their hands so that none could grant licenses without the consent of the other owners. It was alleged that this was an unlawful restraint of trade.

The licenses granted by Consolidated and Massey relat



How a MODERN metal cuts costs here

Creping doctor installed on "Mister Nibroc" at the Berlin, N. H. plant of the Brown Co. "K" Monel is one of the doctor blade materials used on this paper towelling machine, one of the newest and largest of its kind. The creping doctor and "K" Monel creping blades were made and furnished by LODDING ENGINEERING CORP., Worcester, Mass.

Wear and corrosion...worst enemies of your doctor blades . . . *can* be controlled.

The secret of long-lasting, low-maintenance blades lies in using a metal that fits your operating conditions.

"K"* Monel is just such a metal. Consider its unique combination of desirable properties:

- **Non-rusting, highly corrosion-resistant.**
- **Strength and hardness greater than structural steel.**
- **Heat-treatable for maximum properties.**
- **Easy to machine; takes a high polish.**

So much for theory. Now . . . what about practice?

Many of the nation's busiest board and paper mills report outstanding service from "K" Monel doctor blades, in both dry and wet end applications.

"K" Monel blades, operating on modern high-speed paper machines, have given up to *18 times* longer service than blades of other commonly used metals. Less honing and less regrinding are required. Corrosion ceases to be a threat to blade life. Maintenance costs drop sharply.

Right now, diversion to America's defense program has limited the supply of "K" Monel for civilian use.

Ask your nearest source of supply about the current availability of "K" Monel, or for more detailed technical information.

* Reg. U. S. Pat. Off.

THE INTERNATIONAL NICKEL COMPANY, INC.
67 Wall Street, New York 5, N. Y.

EMBLEM OF SERVICE
INCO
TRADE MARK

"K"* MONEL ...FOR MINIMUM MAINTENANCE

to the sale of the product and contain limitations as to the kind of product to be sold and in what territory.

In refutation of the claims made by the Kimberly-Clark Corp., Consolidated Water Power & Paper Co. asserted that the burden of proof of patent invalidity is still upon the defendants, and they deny all the various defenses set up by the defendants. The plaintiff's assert that the disclosure is sufficiently clear to those skilled in the coating art. Also, the plaintiffs asserted that whether or not defendants learned anything from Mr. Massey, if they perform the process claimed then they infringe the patent. Also the plaintiffs asserted that patents which have never been commercialized are "paper patents" and should be restricted to the precise facts disclosed, i.e., no range of equivalents to be allowed to these facts. Also the plaintiffs asserted that with the exception of the Traquair patent, the references produced by the defendants do not relate to the coating field. Moreover the plaintiffs asserted they were entitled in the interpretation of their patent to broad equivalents since it is alleged that plaintiff's patent is a pioneer patent upon which a whole new industry has been founded.—Reprinted from *Paper MillNews*, June 7, 1952.

Consolidated Water Power & Paper Co. has sent the following letter to *Paper Mill News:*

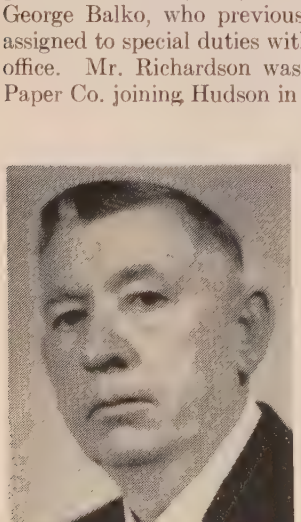
"We note the article in your June 7, 1952 edition concerning patent case of Consolidated Water Power & Paper Co. and Peter J. Massey versus Kimberly Clarke Corp.

"There are a number of technical errors in the lengthy presentation which you made, and we feel that the article is considerably weighted by giving greater emphasis to the arguments of Kimberly-Clark than to our refutations, many of which are completely omitted. This is particularly true of the charge of the violation of the anti-trust laws. The court made no finding to indicate any such violation on the part of Consolidated or Massey. Further, the only patent in suit was U. S. pat. 1,921,368, and this patent expired on Aug. 8, 1950. Consolidated has many important unexpired coating patents covering its on-the-machine coating operations which are not involved in this litigation.

"Since Judge Tehan has not given a formal opinion as yet, it is not possible to say what grounds he relied upon nor what arguments he refuted. We wish to advise you, however, that we have already instructed our attorneys to appeal the decision and to expedite the hearing of the appeal."

Hudson

Joseph L. Richardson has been appointed Resident Manager of the Palatka, Fla., mill of the Hudson Pulp & Paper Co. George Balko, who previously held this position, has been assigned to special duties with the company in the New York office. Mr. Richardson was formerly with the Gulf States Paper Co. joining Hudson in 1946.



Jospeh L. Richardson, Hudson Pulp & Paper Corp.

Harold Holden Eastern Corp.

Eastern Corp.

Harold Holden is now President of the Eastern Corp., Bangor, Me., succeeding Clyde B. Morgan who resigned to become President of Rayonier, Inc. Mr. Holden was formerly Vice-President in Charge of Sales of the Oxford Paper Co.

Mead

Howard Teter, Division Manager of the Mead Corp., Chillicothe, Ohio Division since 1930, has been appointed to the newly created post of Production Manager for White

H. E. Teter The Mead Corp.

O. B. Mason, The Mead Corp.

Paper Operations. Mr. Teter started at 15 with Mead in 1907.

O. B. Mason succeeds Mr. Teter as the Manager of the Chillicothe Division.

John E. Graves, Assistant General Superintendent, is now Production Manager of the Division.

Charles J. Ludwig continues as General Superintendent.

R. C. Bradbury and George H. Sheets will be Assistant to the Division Manager.

Frederick D. Thayer, Jr., has been made Assistant Manager of the Tannin Extract Division at Lynchburg, Va. Mr. Thayer has been with the American Cyanamid Co. at Stamford.

Vulcanized Fiber

Production of vulcanized fiber could be increased 10 million pounds to a total of 70 million pounds annually according to an expansion goal announced by the DPA. Of the 10 million pound expansion program certified by DPA, 6.1 million pounds already are covered by Certificates of Necessity. Vulcanized fiber is a rigid sheet material made by treating a special type of paper with zinc chloride solution and compressing several laminae of the gelatinized paper product to obtain a desired thickness. The material is used for insulation and for the mechanical parts in electrical machinery; as a backing for abrasives; in textile equipment and machinery; and for the manufacture of barrels, tubs, tubes, tote boxes, and ammunition cases.

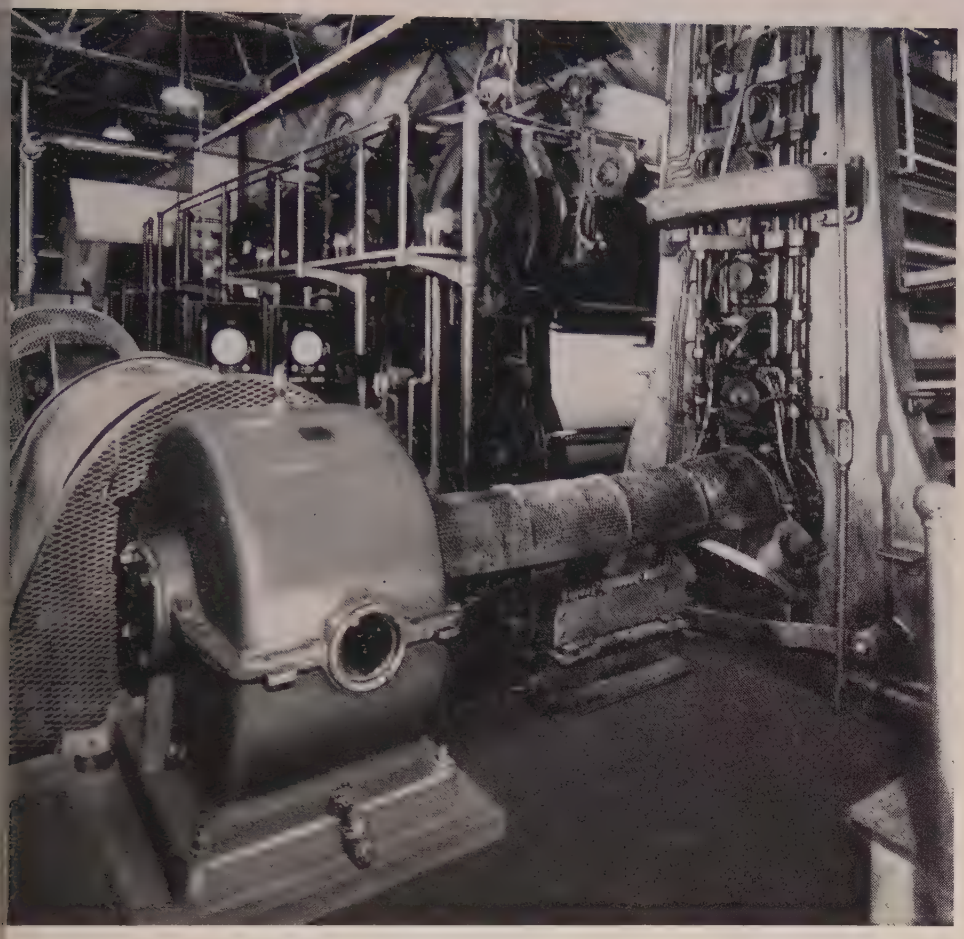
Forest Yield Taxes

The U. S. Department of Agriculture, Washington, D. C., has issued a 50-page circular (No. 899) on "Forest Yield Taxes" by Ralph W. Marquis, Director, Northeastern Forest Experimental Station.

Finnish Mill Record

Oulu Oy's sulphate mill produced 124,114 tons in 1951, registering the European peak attained by a single mill.

Cellulose production reached a postwar record in Finland. Of the total produced (1,385,766 tons), 615,690 tons of sul-



Helps gear units keep their teeth . . .

HEAVIER LOADS and higher speeds brought trouble to 16 large gear units used for driving paper machines at a mid-west paper mill. Under the more punishing conditions of temperature and pressure, the lubricant in the gear sets deteriorated rapidly and had to be replaced every three to four months. Of even greater concern to plant operators was the pitting of gear teeth surfaces—a real threat to uninterrupted operation of paper machines and to plant economy.

Upon the recommendation of a Standard Oil lubrication specialist, the gear cases were drained and cleaned, then filled with STANOGEAR EP Lubricant.

In two years of continuous operation—24 hours a day and 6 days a week—the original fills of STANOGEAR EP have shown no appreciable deterioration and have not needed to be replaced. Plant officials report that the gears show no measurable wear, that pitted surfaces are polished and in excellent condition, and that gear cases are clean.

STANOGEAR EP
REG. U. S. PAT. OFF.
Lubricant

Why not utilize the Standard Oil service employed so successfully by this company? How you can put this service to work for you, quickly and easily, is explained at the right.

Standard Oil Company (Indiana), 910 South Michigan Avenue, Chicago 80, Illinois.

What's YOUR problem?

R. R. Rowlands of Standard Oil's La Crosse, Wisconsin, office is the lubrication specialist who helped this midwest paper mill (see left) solve a serious problem by recommending STANOGEAR EP Lubricant.

Standard's lubrication specialists are located throughout the Midwest. One of them is within easy reach of your plant. You can contact him simply by phoning your local Standard Oil Company (Indiana) office. His experience and special training will help you get maximum benefits from STANOGEAR EP Lubricant and other outstanding products such as:

SUPERLA Greases—Available in a wide range of consistency grades and in both lime-soap and soda-soap types. SUPERLA Greases cover a wide range of operations. These efficient products are comparable in quality with the highest type of special greases.

STANOLITH Greases—Because these unique lithium soap products possess the heat resistant properties of soda-soap greases and the water resistant properties of lime-soap greases, they offer a solution to lubrication problems caused by the presence of both heat and water.

CALUMET Viscous Lubricants—On open gears and wire rope, these greases strongly resist washing and throw-off. Their superior wetting ability affords better coating of gears, better lubrication of wire rope.

STANDARD OIL COMPANY (INDIANA) STANDARD

phite and 417,365 tons of sulphate were sold to domestic and foreign consumers, or 1,033,055 tons in all. Of the quantity sold exports accounted for an aggregate of 979,136 tons.

Rayonier

Ground has been broken for Rayonier, Inc.'s new $25,000,000 pulp mill. When completed early in 1954, the company's fifth plant will produce 250 tons per day of chemically purified wood cellulose to help meet growing world-wide demands for synthetic cellulose products.

Major uses of purified cellulose are the synthetic fibers, rayon and acetate, plastics, film, filament, and cellophane.

Being a growth company serving growth industries, Rayonier in its new plant will use an improved process recently developed by the company's research and engineering division, to manufacture superior grades of wood cellulose, primarily for high tenacity yarn applications.

If necessary, the plant will be able to produce nitration pulp for military explosives as well as cellulose for standard textile yarns.

Approximately 450 people will be employed in the new mill, and an additional 500 will be required by contractors supplying pulpwood.

It is expected that 550 cords of wood a day will be consumed. Part will come from company-owned timberlands, and much will be purchased locally, creating another new market for wood in this southern area.

The Doctortown plant is Rayonier's major development in its $80,000,000 postwar expansion program which began late 1950. It will add 87,000 tons capacity to the company's annual operations.

Syracuse

Michael Szwarc of the University of Manchester, England, has been appointed to the newly established professorship of physical and polymer chemistry at the State University of New York, College of Forestry.

Benchmarks

For packaging paper market analysts: The average man buys 25 razor blades a year, 180 sticks of chewing gum, and 18 pounds of candy. He strikes 3280 matches. There is at least one smoker in 85% of American homes. A man smokes 7.5 packs a week, a woman 5. The population of the U.S.A. on April 1 was 156,405,000.

Olin

John M. Olin, President of Olin Industries, Inc. (including Ecusta Paper Co.), 39 years with his company, presented a 30-year service pin to his brother, Spencer T. Olin, First Vice-President, at the 9th annual meeting of the company's Quarter Century Club. More than 330 of the club's 365 members attended the meeting.

John M. Olin (left), president of Olin Industries, Inc., East Alton, Ill., presents a 30-year service pin to his brother, Spencer T. Olin, first vice-president

Swedish Meeting

The Nordsk Pappersingenjors Kongress will be held in Stockholm, Sweden, on Sept. 9–12, 1952.

Packaging Consultant

Phillip S. Barnhart, formerly of the Westfield River Paper Co., has established a consultation practice at 20 Craiwell Ave., West Springfield, Mass. Mr. Barnhart will specialize in paper packaging problems and is, at present, making a survey to determine the need for an independent packaging laboratory.

Vermont Forests

The Northwest Forest Experiment Station, Upper Darby, Pa., has issued a useful 47-page bulletin, "Forests Statistics for Vermont."

Science Opportunities

The National Association of Manufacturers, 14 W. 49th St., New York 20, N. Y., has distributed 60,000 copies of a booklet "Your Opportunities in Science" that have been sent to high school and college authorities throughout the country. It points out that the wide fields of science-electronics, biology, chemistry, the branches of engineering, research, medicine, etc., in all likelihood offer the best and most chances for young people today.

Pennsalt Paint Maintenance

The Pennsylvania Salt Mfg. Co. has studied coatings that protect against corrosion and has set up the following five points regarding paints:

1. Basically, the dry film thickness is more important than the number of coats, although a minimum of three coats is necessary to insure coverage of pits in the finished surface. In any case, the dry coat has to be at least 5 mils thick.
2. Average direct cost for painting is about 32 cents per square foot. For each coat over three, the cost rises about 17%. Therefore, while a paint that gives a thin film may be cheaper, it may be profitable to use a more expensive paint to get the necessary 5 mils in the first three coats.
3. A regular schedule for inspection, and inspection is a must for the chemical industry. If followed, costs for maintenance painting can be cut to less than 7 cents a square foot.
4. Sandblasting a corroded surface before painting is more effective than going over it with a wire brush. In that way, the life of the coat can be doubled or even quadrupled.
5. Of the industrial paint compositions recommended for general maintenance work, 60% fail rapidly when exposed to conditions met in average chemical plants.

The aforegoing points were developed by Robert Pierce of Pennsalt's Corrosion Engineering Products Dept.

Naval Stores

Naval stores producers are seeking a daily pricing system to replace the outlawed Savannah exchange.

Naval stores include turpentine, rosin, terpene alcohols, and terpene hydrocarbons produced from species of southern pines by (1) tapping line trees (gum naval stores); (2) solvent extracting and steam distilling top wood and stump wood (wood naval stores); (3) destructively distilling dead and stump wood (DD naval stores); and (4) collecting by-products of sulphate wood pulping process (sulphate naval stores).

The Savannah exchange had for years been the market on which the industry based prices. A stable, nationally recognized price is necessary in the case of turpentine, distributed principally in small containers on consignment; and for rosin, at least desirable. The exchange was closed at the be-

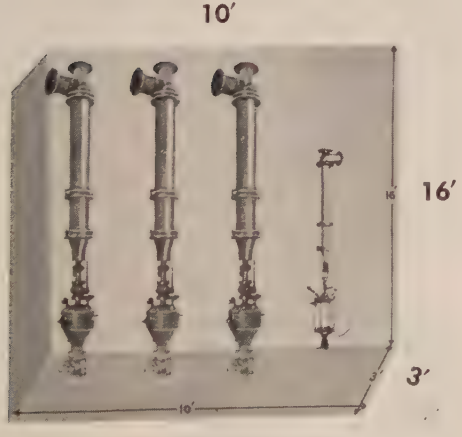
Space a Problem With You, Too?

VORTRAPS

will get you out of a tight spot

. . . handling large volumes of stock with low horse power consumption and a minimum of space requirements.

Fibre Losses - Low
Maintenance - Decreased
Rifflers - Eliminated
Labor - Decreased



The installation of three 10″ and one 4″ Vortraps shown above will handle ***150 TONS*** daily production at 0.75% consistency. (Note the space requirements)

NICHOLS ENGINEERING & RESEARCH CORP.
70 Pine Street — New York 5, N. Y.
40 S. Los Robles Ave., Pasadena 1, Calif.
1477 Sherbrooke St., W., Montreal 25, Canada

hest of the turpentine and rosin farmers who accused the exchange of rigging prices.

Gum products, being classified as agricultural products, are protected by government price supports, level this year being 90% of parity. The current price of gum rosin is about $7.50 per cwt., while that of wood rosin is about $7. Gum rosin is supported at about $7.48 with incidental costs for such as insurance running about 20 cents, so when the price drops below $7.28 it is cheaper for the farmer to put his product under government loans than to sell it. Wood rosin, therefore, is actually setting a price today. A similar situation holds for turpentine.

Normally over 400,000 barrels of rosin should go into paper and paperboard sizes. Wood naval stores do not have a guaranteed floor on price. Of eight producers of wood rosin in the field, three have about 90% of the production capacity. Of these Hercules Powder Co. produces 40%, Newport Industries, 30%, and Crosby Chemicals, 20%. The future of both gum and wood rosin producers will depend largely upon good research efforts.

Westinghouse

F. D. Weatherholt has been appointed Sales Manager of Industrial Products.

Over 100 case histories of applications of Life-Line motors are given in a new 35-page booklet (B-4769) obtainable from Westinghouse Electric Corp., Box 2099, Pittsburgh 30, Pa.

Production Suggestions by Westinghouse rewarded 9000 employees $139,302. The largest award, $2000, was given to G. W. Gunnell, an employee in the Transportation and Generator Division, for suggesting that 2 in. be added to the length of a large sheet of mica, this permitting the cutting of eight pieces of the insulating material instead of six and thereby reducing scrap produced in the operation. The largest single award $5085 was made to Aloysius Estadt in 1948 when he suggested processing mica by machine instead of by hand.

Westinghouse has started construction on a multimillion dollar plant at Raleigh, N. C., to make watt-hour meters.

Joseph S. Parry has been appointed Assistant Manager of the Westinghouse Apparatus Division.

Quality Control

Simon Collier, Director of Quality Control for Johns-Manville Corp., has been elected President of the American Society for Quality Control. Raymond S. Saddoris of A. O. Smith Corp. and J. H. Toulouse of Owens-Illinois Glass Co. were elected Vice-Presidents.

Jackson & Church

David M. Shapleigh has been appointed Sales Engineer in the Zenith Division of Jackson & Church Co., Saginaw, Mich. Mr. Shapleigh was formerly with Paper & Industrial Appliances, Inc., Hercules Powder Co. and the Brown Co.

Swenson Evaporator

Stephen H. Hammond, Chairman of the Board of the Whiting Corp., Harvey, Ill., has announced a greatly expanded advertising and promotion program for 1952. The new program beginning in May is designed to integrate more closely the selling of the various Whiting Divisions, including the Swenson Evaporator Co. The firm of Waldie and Briggs, Inc., has been appointed to handle all advertising and merchandising phases of the expanded program.

Reichhold

Reichhold Chemicals, Inc., has announced the establishment of a new South Atlantic Division at Jacksonville, Fla. This site was selected because it is also a naval stores center, making it a logical spot at which to carry on current Reichhold research into terpene derivatives. The new division will relieve the pressure on the Tuscaloosa, Ala., and Charlotte, N. C., facilities. Annual sales of synthetic resins and allied chemicals by Reichhold last year reached $100,000,000.

Dow

R. H. Boundy, Manager of Dow Chemical Co.'s Plastics Dept. since 1945, has been named head of the company's research activities.

Dow has issued a new bulletin on "Plasticizers." Copies are obtainable from the Plastics Dept., Dow Chemical Co., Midland, Mich.

Hooker

The Hooker Electrochemical Co.'s expansion program calls for an expenditure of $25 million in 1952–53. This will include a $10 million caustic and chlorine plant to be erected at Montague, Mich.; a substantial further expansion of the Tacoma, Wash., chlorine and caustic plant; and additional facilities at Niagara Falls. The H. K. Ferguson Co. of Cleveland has been awarded the construction contracts.

Ingersoll-Rand

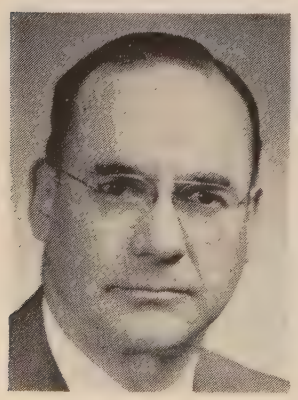
Ingersoll-Rand Co., 11 Broadway, New York 4, N. Y., has issued a new catalog sheet describing its redesign of horizontal, multistage centrifugal pumps for medium pressure application. These have been designated as Class CNTA pumps.

Oliver-United

E. L. Oliver, Jr., has been appointed Executive Vice-President of Oliver United Filters, Inc., Oakland, Calif., New York, and Chicago.

Impco

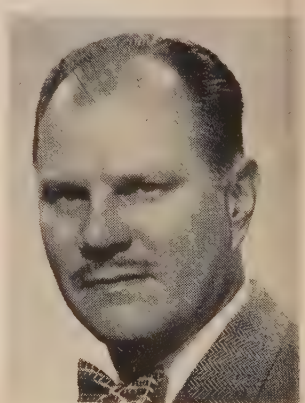
Elmer R. Burling has been elected Executive Vice-President of the Improved Paper Machinery Corp., Nashua, N. H.



D. M. Shapleigh, Jackson & Church Co.

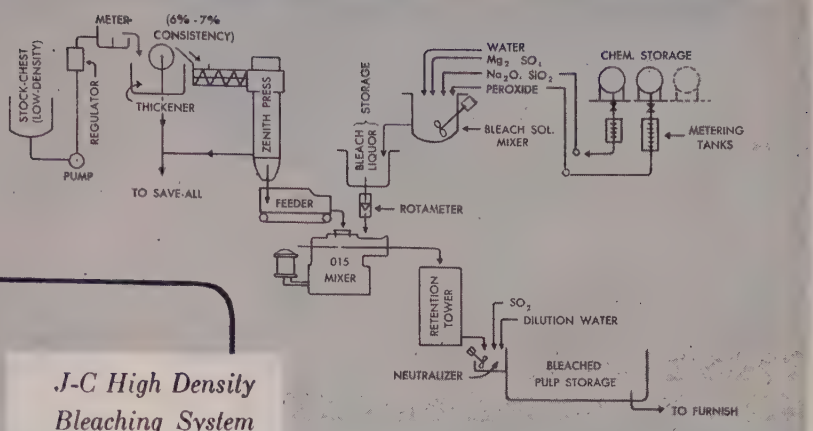
Stephen H. Hammond, Whiting Corp.

E. L. Oliver, Jr. Oliver United Filters, Inc.



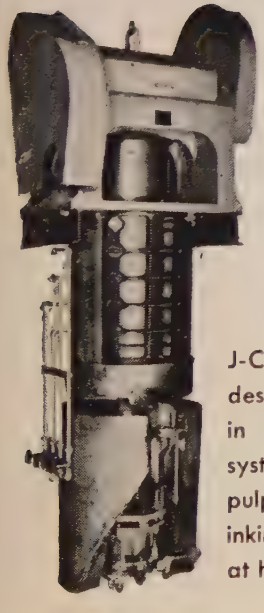
E. R. Burling, Improved Paper Machinery Corp.

Jackson and Church HIGH DENSITY SYSTEM



J-C High Density Bleaching System

USES *NO MORE* PEROXIDE TO BLEACH *HIGHER* TONNAGE TO *SAME* BRIGHTNESS or *SAME* TONNAGE TO *HIGHER* BRIGHTNESS



J-C Zenith Press designed for use in high density systems, and in pulp washing, de-inking and storage at high density.

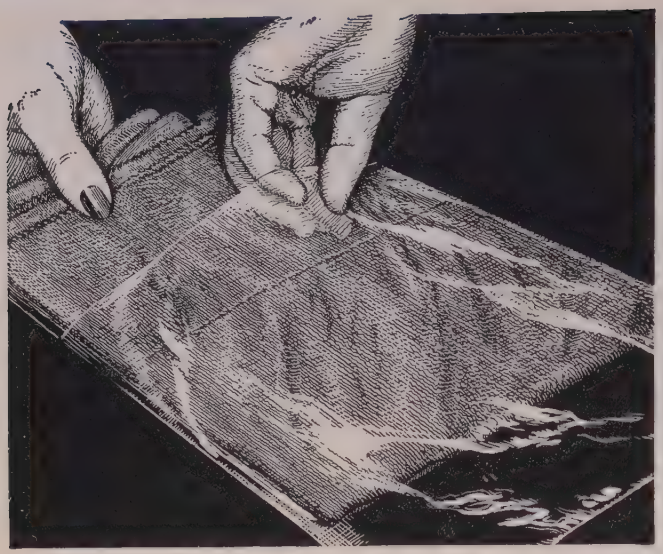
For engineering assistance and actual mill performance data, write Dept. D

The J-C High Density System makes possible bleach consumption savings up to 30%.

HERE ARE ADDITIONAL ADVANTAGES:

High density—35 to 40%
Continuous operation
Fully automatic operation
Selective pressure control
Product of uniform consistency
Moderate first cost
Extremely rugged construction
Available in corrosion resistant alloys
Low power requirement
Minimum floor space
Aids good housekeeping

JACKSON & CHURCH COMPANY · SAGINAW, MICHIGAN



A Finish With Eye Appeal

The glossy surface of this glassine paper attracts the eye—the fine finish is smooth to the touch. Another sale for the right kind of calendering.

Paper-makers throughout the country rely on Butterworth Calender Rolls for top quality finishes at low cost. Their own experience with Butterworth Rolls has convinced them.

Make this simple test. Put a single Butterworth Roll in the stack. See and feel the difference in the finish. Time it in operation. Note the extra hours of service without expensive "down-time".

Butterworth Calender Rolls for coated stock are made of fine grade long staple cotton. Made for super and glassine from high quality paper. We can also refill your present rolls. Write us of your needs.

For full information, write or call H. W. Butterworth & Sons Company, Bethayres, Pennsylvania – 187 Westminster Street, Providence, R. I. : : 1211 Johnston Building, Charlotte, N. C.

Butterworth
CALENDER ROLLS

ALLIS-CHALMERS

Allis-Chalmers Mfg. Co., 828 S. 70th St., Milwaukee, Wis., has issued a new bulletin on "Squirrel-Cage Induction Motors" (51B6210D) giving construction features, ratings, and dimensions.

FIBERGLAS

Owens-Corning Fiberglas Corp. of Toledo, Ohio, recently held a conference for paper and tape manufacturers at Bedford, Pa. Representatives of 40 paper converting companies

A mock-up of a laminating machine demonstrates methods of introducing Fiberglas yarns into reinforced paper. Left to right are Fred Shepherd of Ruberoid Co. (facing camera), J. L. Klecka, also of Ruberoid, Bradley Higbee of Adams Paper Co., John Halsey of Albermarle Paper Mfg. Co. and H. C. Crandall of Mosinee Paper Mills Co.

attended and visited the fiberglas textile plant at Huntington, Pa. Demonstrations were made showing the methods of applying fiberglas yarn and scrim fabric as paper reinforcement.

CHEMICAL SHOW

All floor space for 250 exhibitions has been sold out for the National Chemical Exposition to be held at the Coliseum on Sept. 9–13, 1952.

TRIST COATINGS

The following officers and directors of Trist Paper Coatings, Inc., were elected recently: Chairman, A. Ronald Trist; President, Eli de Vries; Vice-President, David N. Finnie; Secretary-Treasurer, Charles Sumner Barton. The headquarters is located at 65 Tainter St., Worcester, Mass.

BLACK-CLAWSON (SHARTLE & DILTS)

Otto W. Hein of Black-Clawson Co., has been elected Director of Black-Clawson International Ltd., London, England. Black-Clawson International with manufacturing facilities at W. A. Baker, Ltd., Newport, England, has concentrated its efforts on the manufacture of Shartle Bros. Division line of stock preparation equipment.

BIRD MACHINE

Sven Faklgren who will devote considerable time to the efforts of the Bird Machine Co.'s West Coast office will continue to be located at South Walpole, Mass.

GENERAL ELECTRIC

The General Electric Co., Schenectady, 5 N. Y., has issued a "Progressive Mechanization Manual" (GEA - 5789). The program is designed for use by large industrial firms.

General Electric will build a multimillion dollar transformer manufacturing plant at Rome, Ga. It will be ready by mid 1953. The Pittsfield, Mass., plant will continue to be the main plant for manufacturing transformers.

SOLVAY

Effective June 1, the Solvay Process Division and the Solvay Sales Division were consolidated and are now known as the Solvay Process Division, Allied Chemical & Dye Corp. This division will be responsible for the production and sales of alkali, chlorine, and related products. The executive sales office has moved from 40 Rector St. to 61 Broadway, New York, N. Y.

Solvay Technical and Engineering Service has just issued the Third Edition of Bulletin No. 5 on "Soda Ash—Properties, Loading and Unloading, Handling and Storage."

GRATON & KNIGHT

Graton & Knight Co., 356 Franklin St., Worcester 4, Mass., now issues its Grako (r) Sprazon, belt dressing in 12-oz. spray containers. The spray is directed at the smaller pulley face where slippage is most likely to occur.

SANDY HILL

Sandy Hill Iron & Brass Works, Hudson Falls, N. Y., has issued a catalog of Modern Pulp and Paper Machinery. An excellent job.

CYANAMID

Palmer W. Griffith, West Coast Technical Sales Director of the American Cyanamid Co., received the 11th annual John Wesley Hyatt Award for achievement of wide importance to the plastics industry by the Society of the Plastics Industry for his development of melamine resins.

WITCO

A newly revised and expanded Technical Service Report covering mildewing agents with respect to government agents has been made available by the Witco Chemical Co., 295 Madison Ave., New York 17, N. Y.

PLASKON

Plaskon Division, Libbey-Owens-Ford Glass Co., Toledo, Ohio, has issued a new brochure on Plaskon products "Reducing Wet-Strengthening Costs with Plaskon Aminoplast."

AMERICAN RESINOUS CHEMICALS

American Resinous Chemicals Corp., Peabody, Mass., has issued a data sheet C-66 which describes a number of formulas for adhesives for use on paper, foil, etc.

American Monomer Co., Leominster, Mass., has issued data sheets on its new product Lemol (polyvinyl alcohol) for use in adhesives.

HERCULES

A small folder describing the advantages of using Kymene to obtain better wet-strength paper bags is available from the Hercules Powder Co., Wilmington, Del. In addition to a sample of bag paper containing Kymene taken from a connected run in a mill, the folder contains test data.

Charles A. Grant has been appointed Chicago District Sales Manager of Hercules Cellulose Products Dept.

A revised edition of the technical booklet about S S Nitrocellulose, has been issued by Hercules. It includes 20 charts which illustrate studies in solubility of S S Nitrocelluloses.

INTERNATIONAL NICKEL

International Nickel Co., 67 Wall St., New York 5, N. Y., has issued two new bulletins: "Some Properties of Low Carbon 8½% Nickel Steel" (12 pages) and "Mechanical Properties of Nickel Deposits" (12 pages).

YOU CAN RELY ON PERMUTIT EQUIPMENT

Permutit equipment gives you *complete control* of water quality. Color, iron, dirt and other process water impurities that streak and spot pulp and paper products are removed. The result is *improved* quality which enables you to satisfy customer demands for high-grade papers.

Write for full information about Permutit Precipitator—reliable equipment assuring you of chemically correct water at all times. The Permutit Company, Dept. TAP, 330 West 42nd Street, New York 36, N. Y., or to the Permutit Company of Canada, Ltd., 6975 Jeanne Mance Street, Montreal.

PERMUTIT CONTROLLED PROCESS WATER MEANS:

- **Clean, high quality paper products.**
- **Higher production—lower production costs.**
- **More sales... bigger profits.**

PERMUTIT®

WATER CONDITIONING HEADQUARTERS FOR 40 YEARS

NEARLY
300
BAUER REFINERS IN USE ON SEMI-CHEMICAL JOBS ALONE
(as of May 1, 1952)

Four of the nine Bauer Refiners in a board mill where these machines do all the refining on semi-chemically cooked chips.

Equipment for Pulp and Paper Mills

Bauer

Double-Disk Refiners
Single-Disk Refiners
Mead Laboratory Beater
McNett Fiber Classifier
Attrition Mills
Hammer Mills
Laboratory Mills
Magnetic Separators

Ask for literature and data

THE Bauer BROS. CO.

Established 1878

1758 SHERIDAN AVE.
SPRINGFIELD, OHIO

VULCAN

The Vulcan Copper & Supply Co., Cincinnati, Ohio, has opened a new office in the Chrysler Bldg. East, 161 E. 42nd St., New York, N. Y.

V. D. Drummond, Vulcan Copper & Supply Co.

Raphael Katzen, Vulcan Copper & Supply Co.

V. D. Drummond, formerly Manager of Vulcan Engineering Division, is Manager of the New York office. Raphael Katzen is now Manager of Vulcan's Engineering Division.

G.A.T.C.

The General American Transportation Co., 135 S. La Salle St., Chicago 90, Ill., has issued a new brochure on "Filters" which describes the products of the company with a guide to selection.

RELIANCE

The new Euclid plant of the Reliance Electric & Engineering Co. was opened for inspection by business paper editors in May. The new supermodern structure is located on a site comprising 62 acres, providing for future expansion five times as large as the present plant. The plant floor space occupies 130,000 sq. ft. and was built at a cost of $1,800,000. The building is under positive pressure to exclude dirt and provision is made for six changes of air per hour, three in Winter in the factory section.

View of the new Euclid plant of the Reliance Electric & Engineering Co.

Design-wise, the new plant is distinctive in many respects. Planned for multifunctional usage, its production facilities are readily convertible to meet the changing requirements which may be brought about by new product developments. Layout is likewise flexible to meet anticipated needs of future expansion.

The plant enables Reliance to centralize conveniently, under one roof at one location, the company's diverse tech-

Use of module sections in engineering offices of new Euclid plant of Reliance substantially reduces lateral noise transmission. Noise is further reduced by the suspended, sound-absorbing ceiling of acoustical tile. By these two measures, the general noise level is kept well under the limits required for concentration on detailed work

nical service functions and facilities required in the design, development, production, repair, and service of motors and electronic-type controls for adjustable speed drives. Specifically these are: (1) engineering, research, and development laboratories; (2) manufacture of electronic equipment and motor drive controls, output of which is being stepped up 100% for both defense and civilian applications; (3) renewal parts, motor repair, and related customer service activities; (4) design, development, and manufacture of specialized tools, dies, jigs, fixtures, etc., required by Reliance's production departments in Cleveland and Ashtabula.

Extremely wide column spacing (40 by 50 ft.) is a feature of the factory area. Low bay areas have a 15-ft. clearance under trusses, the high bay area, a 24-ft. clearance from floor to trusses.

Materials handling has come in for particular attention with the liberal use of roller conveyor sections, a prime consideration in moving one of the company's chief products, VS

Limitation of windows to four rows of sash high up beneath plant roof permits full 12 ft. clear walls for present storage of supplies, possible future location of production machinery. Plant transportation has been kept flexible through use of a variety of battery-operated trucks, some of which, as shown here, are specially designed for handling storage of even sizable objects at considerable heights

Clean 'Im Out!
Clean 'Im Out!
Clean 'Im Out!

That's the only satisfactory answer to SLIME.

Use all the preventive measures you want . . . and SLIME will continue to creep in. The cain it raises depends on your housekeeping! Let it pile up, and you have a major operation on your hands. Clean it out regularly, and your slime troubles are never serious.

Do it THOROUGHLY and EASILY!

the economical Magnus way. Magnus Slime Remover is a powerful alkaline cleaner equipped with ultra-fast wetting action. It penetrates deep into the heart of slime deposits wherever they are, loosening their bond to metal surfaces and removing them without need for a lot of hand work. Magnus Slime Remover also kills fungi and bacteria, particularly in hard-to-reach areas where they remain to speed up slime formation when ordinary cleaning methods are used.

SCHEDULE THE JOB!

Don't wait for SLIME to accumulate and *then* clean. Use Magnus Slime Remover on a regular scheduled cleaning routine. You'll find Magnus Slime Remover is the most economical cleaner you can use, based on results!

LOOK IT UP IN THE HANDBOOK

Slime Control through better housekeeping is covered on pages 4 to 8, and 19 to 22 of the Magnus Paper Mill Cleaning Handbook. Look it up. If you've misplaced your copy — ask for another one!

MAGNUS CHEMICAL CO. • 104 South Ave., Garwood, N. J.
In Canada—Magnus Chemicals, Ltd., Montreal.
Service representatives in principal cities.

Magnus
CLEANERS • EQUIPMENT • METHODS

COMPLETE ENGINEERING AND CONSTRUCTION SERVICES FOR THE PULP AND PAPER INDUSTRY

If you have a problem involving any of the following, you can get immediate and experienced help from EBASCO engineers and consultants:

- Design and Construction of Pulp and Paper Mills
- Steam and Hydro-Electric Power Plants
- Power Studies
- Reports and Appraisals
- Recovery Plants, Extensions and Alterations

For complete details on EBASCO's various services send for "The Inside Story of Outside Help." Address Dept. P, Two Rector Street, New York 6, N. Y.

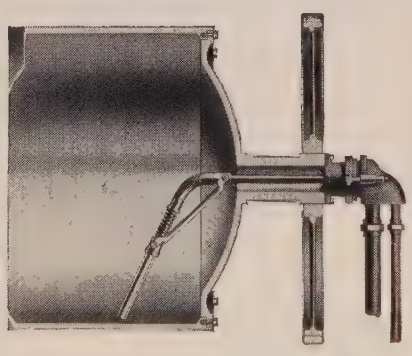
ENGINEERS · CONSTRUCTORS · EBASCO · BUSINESS CONSULTANTS

EBASCO SERVICES
INCORPORATED
NEW YORK • CHICAGO • WASHINGTON, D. C.

Double Barrel Reason

Reaction of ¾" Auto-Flex in 48" dia. dryer. Very low leverage.

Reaction of ¾" ordinary bent pipe. Syphon in 48" dia. dryer. High leverage.



for using Midwest Auto-Flex Syphons

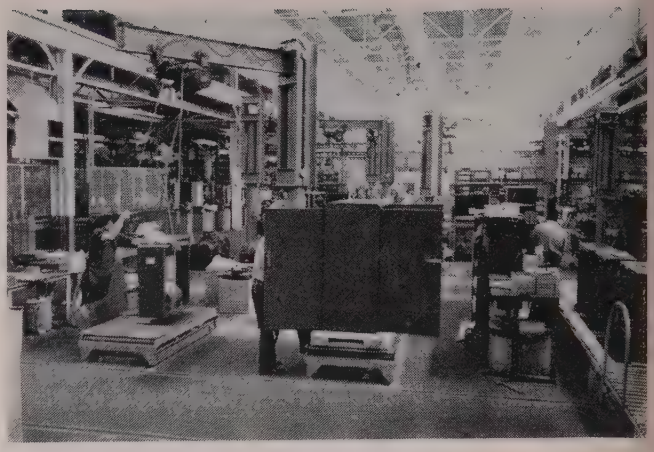
Midwest Auto-Flex syphons have been on the market for years and many thousands are in service for two very excellent reasons—they hold their position—they wear indefinitely.

1. The spring and brace holds the end of the pipe at the proper distance from the bottom of the dryer. It cannot possibly take any other position — hence, proper water and air removal.

2. The cantilever leverage exerted is lower than with bent pipe syphons, only 170# vs 354#. It clears instead of dragging on the journal and rapidly wearing out.

Order a get acquainted dozen. Performance as stated guaranteed.

Midwest-Fulton Machine Company Dayton • Ohio



Other materials handling facilities include profusion of jib cranes equipped with electrical hoists. A number of roller conveyor lines have also been installed. Since many operations are performed on parts in transit without removing them from line, two heights of conveyor track, depending on nature of materials handling, have been installed so employees can always work without stopping

drives, from first assembly operations to final crating and delivery to the shipping dock. This progression is made possible by means of roller conveyor sections mounted on transfer cars for movement between conveyor lines and by roller conveyor sections mounted on electrically operated elevators for transfer between conveyors at different levels.

Electric power at 33,000 volts is brought overhead by the local utility to a nearby outdoor substation for initial reduction to 4160 volts for underground transmission into the plant, where it is further stepped down by a 1000-kva. Askarel-cooled transformer to an a.c. 2300-volt feeder to operate motor-generator sets supplying lower laboratory and test voltages. Voltages of 480, 440, and 220 are also needed for other plant requirements.

Lighting is furnished through spot installations of dry-type transformers and controlled by area control lighting panels. Slimline, single pin, instant start, color cathode fluorescent fixtures installed in 8-ft. lengths suspended from the roof provide approximately 35 to 40-ft. candles in factory areas and from 60 to 65-ft. candles for general office areas.

A combination automatic and forced air system is used in heating and ventilating. It keeps the pressure inside slightly above the atmosphere outside. The conventional boiler room or central heating plant is absent. In its place are two modern gas-fired steam boilers. Installed in a low gallery over the office area adjoining the factory, they provide 5-lb. steam for coils for three blower units which heat offices and service areas, and also supply steam for heating the hot water circulated through fin-type wall radiators installed beneath windows.

Factory areas are heated and ventilated by 11 direct-fired, horizontal-type unit heaters ranging in output from 500,000 to 1,250,000 B.t.u. In summer months, oxivane axial-flow fans, installed parallel to eight main units in factory areas, are used to double the air flow.

Considerable new equipment to facilitate research and testing has been installed, including a 250-hp. dynamotor backed up by a 450-hp. synchronous motor-generator set with a 300-kw. d.c. generator and a 312-kva. alternator. Through a plug board, this unit can be connected to give its full power output at voltages of 220, 440, 660 or 1320 on 60 cycles. With the d.c. generator serving as a driving motor any frequency up to 60 cycles can be contained. Line capacity of 1000 kva. at 440 volts is available for a 60-cycle a.c. motor testing.

The laboratory contains a special sound-conditioned room. Radio and acoustically quiet, it is used for the study of radio interference problems as well as those encountered in main-

fining an acceptable motor noise level in the face of the ever-creasing ratio of power output to motor size.

Much equipment simulation is accomplished with the ngineering Dept.'s linear analog computer. However, there le instances when specialized drives, beyond the range of this mputer, must be tested, using mock-ups of the actual equip-ent. All such testing has the common objective of finding oblems before engineered drives are built and shipped.

The ever-broadening application of the Reliance all-elec-ic, adjustable-speed drive has increased the need for a totally closed motor which by virtue of special cooling provisions uld be built in relatively large horsepower ratings for those tuations where it is necessary for the motor to be given extra otection from atmospheric conditions.

In many instances, especially when the drive motor must be otected from bad atmospheric conditions, the unit must be closed; and, when enclosed, must be properly cooled within self, preferably by clean air.

To meet this type of service, Reliance has developed what ay be termed a totally enclosed dual-cooled motor, equipped ith a heat exchanger as an integral part of its own frame. 1.5-hp., 3-phase, 60-cycle motor, built into the heat ex-hanger, runs at 3600 f.p.m. to drive internal and external ans independently of the main motor itself.

The heat exchanger sections are cast aluminum with both nternal and external fins for maximum heat dissipation. Air velocity through the internal sections of the exchanger is 2900 f.p.m. In the external path between the cover and the exchange bank the air is pumped at 3300 f.p.m.

Announced at the inspection was the development of a new d.c. motor of low inertia (small diameter armature) and high flux (strong field) designed to meet the extraordinary fast reversing requirements of the drives for many of today's high-production machines.

The new motor, based on the Reliance type T heavy-duty design, makes use of two electronic circuits to impose, briefly, several times normal voltage on both motor armature and field. Controlled within predetermined current limits, these forcing circuits make it possible while maintaining maximum torque to reverse the motor more rapidly than has heretofore been achieved by anyone.

Another announcement of a new product was the new floating worm gearmotor. This motor makes it possible to apply power directly to the roller shaft. This has been achieved through designing the unit so that the shaft of the hollow worm gear can be slipped over an extension of the shaft of the roller. Use of a suitable restraining device keeps the motor from rotating around the roller shaft.

The function of the worm gear is to produce the necessary torque at low speeds, lower than can be maintained satisfactorily by a direct motor drive.

Maine

Twenty water colors are being exhibited at the University of Maine Art Gallery from June 1 to August 15 by Laura J. A. Neese of Beloit, Wis. Included among the paintings are some views of the buildings and machines of the Beloit Iron Works.

The University of Maine Pulp and Paper Foundation has received gifts lately from the National Association of Waste Materials Dealers, Scott Paper Co., Eastern Corp., Improved Paper Machinery Corp., Samuel M. Langston Co., Cameron Machine Co., Denison Mfg. Co., and the Niagara Alkali Co.

Ketchikan

The construction of the 45 million dollar mill of the Ketchikan Pulp Co. is under way and should be completed within 2 years. Wood for the manufacture of high-grade dissolving pulp will come from the Tongass National Forest, the contract providing for a 50-year supply of timber for a mill of 525 tons daily capacity. The initial mill will produce 300 tons

ANSUL
SULFUR DIOXIDE
Cuts Costs
IN
PAPER PRODUCTION
TECHNICAL BULLETINS TELL HOW

In the paper industry the use of Ansul Sulfur Dioxide . . . *plus the services of the Ansul staff of chemical engineers* . . . has resulted in new economies, better controls and improved quality in a variety of applications.

GROUNDWOOD PULP MANUFACTURERS use Ansul Liquid Sulfur Dioxide as a neutralizer in their peroxide bleaching operation. It is safe and economical to use . . . *and improves brightness, too!*

NEUTRAL SULFITE PULP MANUFACTURERS have avoided costly delays and shut-downs by using Ansul Liquid Sulfur Dioxide to manufacture their own sodium or ammonium sulfite.

And the Ansul "Controlled SO_2 System" eliminates the need for burner gas, decreases costs and provides efficiencies well worth investigating.

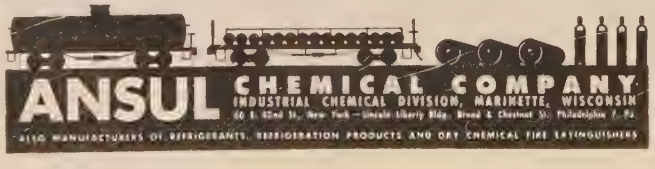
Ask for information relating to your operations. Valuable data will be sent to you promptly and Ansul's chemical engineering staff will cooperate with you willingly and without cost or obligation.

ANSUL LIQUID SULFUR DIOXIDE

. . . is the quality standard of the industry . . . 99.9+% (by weight) pure.

. . . is shipped in 150-lb. cylinders, 2000-lb. ton drums 30,000-lb. tank trucks, 40,000 and 60,000-lb. tank cars.

. . . is available for immediate shipment, in any desired quantity to any domestic and approved foreign destination.



CONVERT STARCH

with

AMYLIQ

A Highly Purified Enzyme Product

1. Costs less than pre-converted starches and does an equivalent job.

2. Simplifies purchasing and inventory control because only one starch is needed with AMYLIQ.

WALLERSTEIN COMPANY, INC. 180 MADISON AVE., NEW YORK

Look to the Source!

When You Use Sodium Chlorate From Oldbury . . . you may rely upon the reputation of your source of supply as absolute assurance of the product *quality* and *uniformity* that you must have to maintain and advance your own product standards and customer services.

OLDBURY

ELECTRO-CHEMICAL COMPANY

Plant and Main Office:
NIAGARA FALLS, NEW YORK

New York Office:
19 RECTOR STREET, NEW YORK 6, N.Y.

daily. It is a joint enterprise of the Puget Sound Pulp & Timber Co. and the American Viscose Co.

PRINTING INK

Ernest Gamble, Jr., has been named Assistant Director of the National Printing Ink Research Institute at Lehigh University, Bethlehem, Pa.

DIRECT MAIL

The Direct Mail Advertising Association reports an 11% increase for direct mail advertising for the first quarter of 1952 compared with the same quarter in 1951. The 1951 volume was up 14% over 1950.

WASTEPAPER

Receipts of wastepaper for 1952 through March totaled 1,933,000 tons, a decline of 32% under receipts for the similar months in 1951.

Wastepaper Consumptions (Tons)

Year	*For paperboard*	*For roofing and building materials*	*For paper*	*Total*
1946	5,739,300	431,700	1,108,600	7,279,600
1947	6,303,100	536,600	1,169,300	8,009,000
1948	5,943,300	566,000	1,139,700	7,649,000
1949	5,090,700	520,100	988,800	6,599,600
1950	6,504,700	466,300	932,600	7,903,600
1951	6,963,502[a]	771,704[a]	1,343,673[a]	9,078,879

[a] Estimated.

Wastepaper consumption for the first quarter amounted to 1,941,000 tons, a decline of 25% under consumption for similar months in 1951.

Ratio of Wastepaper Recovery to Board and Paper Consumption

Year	*Board and paper consumption, tons*	*Mill wastepaper receipts, tons*	*Ratio of wastepaper recovery, %*
1946	22,509,821	7,487,000	33.3
1947	24,756,500	7,875,000	31.9
1948	26,105,700	7,670,000	29 4
1949	24,751,900	6,522,000	26.3
1950	28,998,400	7,895,000	27 2
1951	30,703,000	9.252,000	30.1

National wastepaper consumption, during the month of April, dropped about 8.5% below that of March.

Exports of Wastepaper (Tons)

Year	*News grades*	*All other grades*	*Total*
1946	14,587	59,222	73,809
1947	30,883	120,162	151,045
1948	23,080	134,274	157,354
1949	74,265	42,932	117,197
1950	68,495	51,206	119,701
1951	102,383	104,055	206,438

At the present rate of consumption (first third of year), 1952 will be a better year than previous ones except 1951, 1950, 1948, and 1947.

The greatest increase in the amount of wastepaper consumed last year over 1950 was by the roofing and building materials mills. Last year their consumption averaged 63,600 tons per month. This year, the rate, based on the first two months, is 52,600 tons per month.

Numerous requests for statistical information regarding the recovery and consumption of wastepaper during industry's record-breaking year of 1951 have resulted in the assembling of information from the U. S. Department of Commerce, the

Per Capita Production, Paperboard and Paper

Year	U. S. population	Paperboard, tons	Pounds per capita	Paper, tons	Pounds per capita	Total pounds per capita
1946	141,228,693	9,504,200	135.0	9,773,400	138.4	273.4
1947	144,881,000	10,408,900	143.7	10,705,100	147.8	291.5
1948	148,711,000	10,774,700	144.9	11,153,600	150.0	294.9
1949	150,998,000	9,956,200	131.9	10,371,000	137.4	269.3
1950	153,490,000	12,206,500	159.1	12,093,700	157.6	316.7
1951	154,353,000	13,064,000	169.3	13,022,000	168.7	338.0

American Paper & Pulp Association, and other sources and the following compilation of tables to supply one concisely with the complete picture. Per capita figures are based on a U. S. population of 154,353,000 in 1951.

Production, Paperboard and Paper

Paper production, all types, tons	13,022,000
Per capita, lb.	168.7
Paperboard production, all types, tons	13,064,000
Per capita, lb.	169.3
Total paper and paperboard production, tons	26,086,000
Per capita production, 1951, lb.	338

Consumption, Paperboard and Paper

Total U. S. production, paper and board, tons	26,086,000
Total imports to U. S., paper and board, tons	5,144,000
Total production and imports, tons	31,230,000
Total exports, paper and board, tons	527,000
Total U. S. consumption, paper and board, tons	30,703,000
Average per capita consumption, 1951, lb.	397.8

Recovery of Wastepaper

U. S. total mill receipts of wastepaper, tons	9,252,000
Wastepaper exported from U. S., tons	206,438
Wastepaper salvaged in U. S. in 1951, tons	9,458,438
Wastepaper salvaged per capita, 1951, lb.	122.5
Ratio of recovery of wastepaper to total consumption of paper and paperboard, 1951, %	30.8

Consumption of Wastepaper

Total U. S. consumption of wastepaper, tons	9,078,879
*Consumed by board mills, tons	6,963,502
*Consumed by paper mills, tons	1,343,673
*Consumed by roofing and building material mills, tons	771,704

*Estimated.

U. S. Wastepaper Inventories

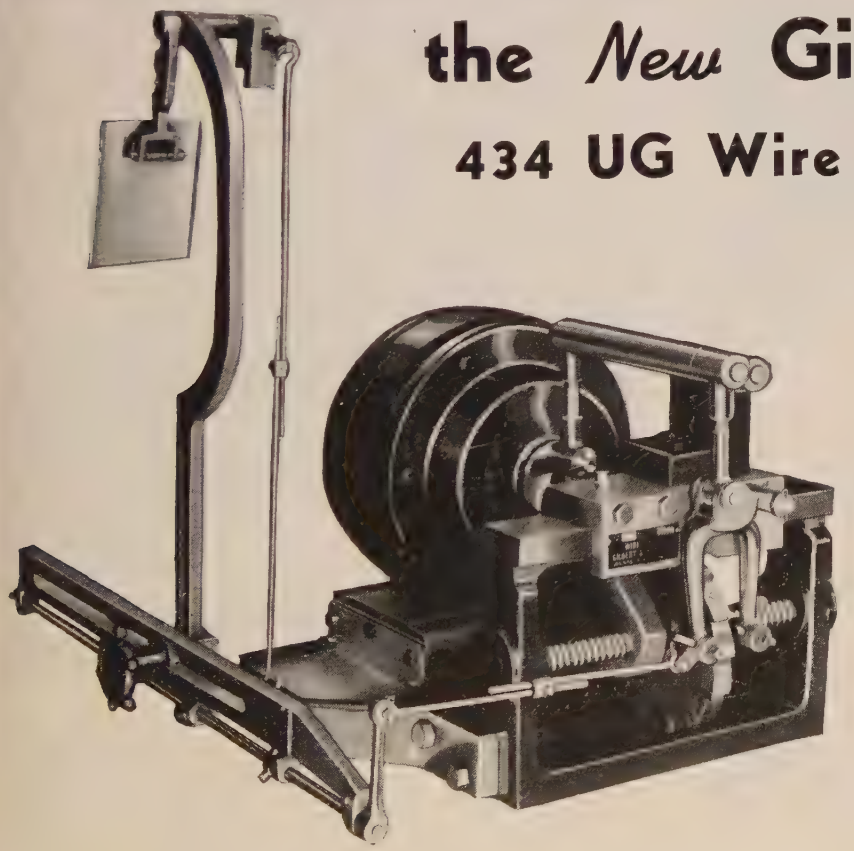
At year end, 1950, tons	385,000
At year end, 1951 tons	588,000

Badger

The Badger Paper Mills, Peshtigo, Wis., has installed a 100,000-gal. storage tank for sulphite spent liquor. This is being used as a supply tank for tank trucks that haul this material away for 16 hours a day, for use as roadbinder, and for disposal in the company's ponding operation. The 3000-gal. trucks are loaded in less than 5 minutes.

Lowell Textile

The first two degrees to be awarded in Paper Engineering by the Lowell Textile Institute, Lowell, Mass., were granted on June 9 to Vernon J. Beaulier and Alfred K. Hobbs. Mr. Beaulier received the Bachelor's degree. Mr. Hobbs, a 1950 graduate of the University of Maine, received a Master's degree and will become a member of the Institute's staff as an instructor.



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Girdling Tool

The Northeast Pulp Research Center has developed a special tool for girdling trees that are to be treated for bark removal. It is generally limited to bark not over $^5/_8$ in. in thickness, and the present size permits the removal of a 4-in. strip of bark, without cutting into the sapwood. Distributed by Woodlands Equipment, P.O. Box 334, Gorham, N. H.; price $45. Another convenient unit, a back pack tank and pressure unit for applying the poison to the girdled trees, can be obtained for about $45.

Valite

Research into the processing of bagasse for commercial uses was started in 1936 by Valentine Sugars. Recently the affiliated Valite Corp. furnished samples of 100% bagasse newsprint-type paper, including all the pith, to Louisiana sugar-belt newspapers for printing tests. Executive offices of the Valite Corp. are located in the Whitney Bldg., New Orleans, La. The manufacturing facilities are located at Lockport, La.

W. R. Grace

At a press conference sponsored by the W. R. Grace Co., samples of paper made from sugar cane bagasse were exhibited. The samples included printed copies of *La Prensa*, a Peruvian newspaper made of 100% bagasse. Speakers for the Grace Co. advised that the paper was of high quality and although it cannot, at present, be made for sale competitively in the United States market, it can be manufactured and sold successfully in countries that have large sugar cane centrales.

W. R. Grace has been making bagasse papers for the past 12 years at its Paramonga sugar plantation.

Employing the huge bagasse resources of a 14,000-acre sugar cane plantation, Paramonga's Paper Mill has succeeded in increasing production more than 500% in its first 12 years of operation. Total output of paper products in 1940 was around 3000 tons. By 1951 the figure had jumped to 19,000 tons, and facilities are still being expanded.

The over-all percentage of bagasse used in the production of the various grades of paper has increased sharply over the years. In 1940 bagasse accounted for 21% of the total pulp

With the bagasse resources of a 14,000-acre sugar plantation at its disposal, the Paramonga Paper Mill turns out a steadily increasing quantity of paper manufactured from a pulp containing a high percentage of bagasse—the fibrous residue after the sugar has been removed from cane. The mill is operated by a subsidiary of W. R. Grace & Co., international industrial and trading concern. Several varieties of paper are made with a 100% bagasse content while others utilize slightly lesser amounts. The average bagasse content of all Paramonga paper products is about 70%

Thousands of bags of refined sugar are stored in the open air at Hacienda Paramonga, sugar estate and industrial center on the north coast of Peru, which is operated by a subsdiary of W. R. Grace & Co. The sugar is packed for shipment to domestic and foreign markets in multiwall bags made with a high bagasse content pulp. Paper bags for shiping sugar are preferred over cotton because they keep the contents uncontaminated and give more protection.

processed at Paramonga, but at present the figure is closer to 70. Certain types of paper, i.e., newsprint and printing papers, are made from 100% semibleached bagasse pulp; paperboards, utilizing from 60 to 80%, are generally next highest in bagasse content. In other qualities the proportion varies from 45% in multiwall kraft bag paper up to a maximum of 75 to 85% in sulphite wrappings.

Strong, moisture-resistant multiwalled bags from 2 to 6-ply as well as sturdy corrugated cartons used in packaging Peruvian fish and other goods for export or domestic sale are imprinted at Paramonga with the customer's trademark. Small paper sacks range in size from the 1 to 25-lb. capacity.

Although paper products of all types except newsprint are exported in limited amounts to Bolivia, Ecuador, and Colombia, most of Paramonga's output is absorbed by the Peruvian market. The Grace subsidiary supplies some newsprint to prominent dailies including *El Comercio* and *La Prensa* of Lima; heavy-duty bags to the domestic sugar, cement, lime, quartz, minerals, and flour industries; and kraft (brown) wrapping papers, both plain and striped, for use in the nation's drug and department stores, as well as filling other essential paper requirements.

The Paramonga process consists first of washing and screening bagasse to remove the pithy material, cooking the remaining fibrous pulp for the time required to produce distinct paper types, and lastly refining to the degree most suited for the production of the various grades.

The method, evolved after long and painstaking experimentation, was developed from tests carried on under the direction of G. J. Lipscomb, retired Vice-President, formerly in charge of the Industrial Department, from 1932 to 1934 in the Research Laboratory of Hacienda Cartavio, another sugar estate operated by a *Casa Grace* subsidiary in Peru. The work begun at Cartavio was continued in a research laboratory in the United States, and in 1935 when progress appeared to warrant a commercial trial in the U. S., the idle Stony Brook Mill of R. B. McEwan & Son of Whippany, N. J., was leased for the purpose.

Some 2000 tons of bagasse were imported from Peru and processed with the pulp and papermaking equipment available at Whippany. The test was a marked success with nine-point board, found entirely acceptable to United States corrugated box manufacturers, accounting for almost the entire

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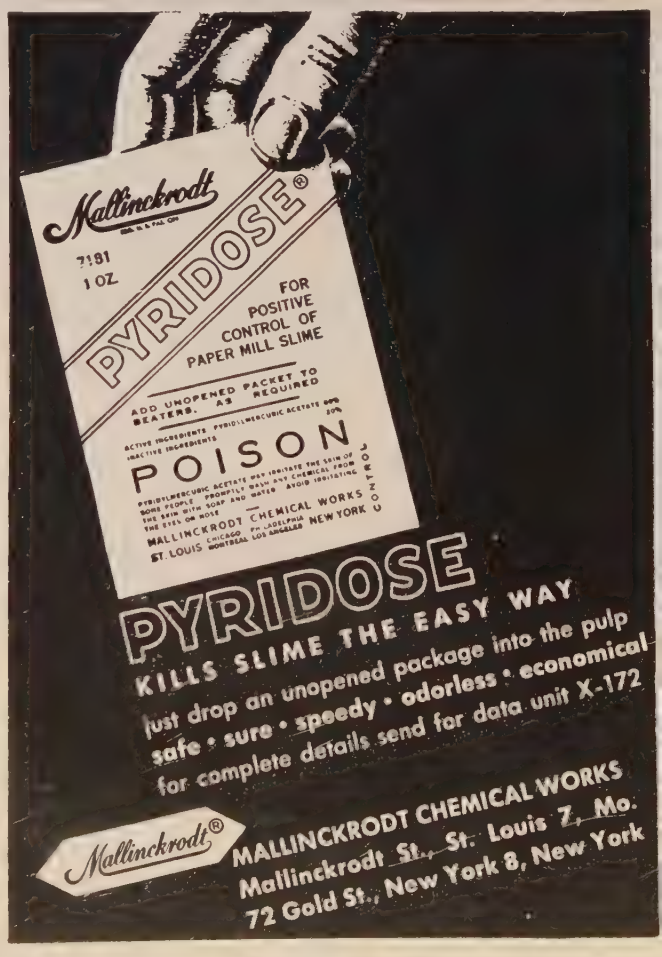
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Sandy Hill Iron & Brass Works of Hudson Falls, N. Y. has entered into an agreement with W. R. Grace & Co., international industrial and trading concern, whereby the former will manufacture the special machinery needed for producing paper from bagasse. This view shows a paper machine in a paper mill at Montevideo (Uruguay) which uses a straw-base pulp: the equipment for this mill was produced by the Sandy Hill factory

production. Encouraged by results, the company launched a market and plant site study in Peru. Plans for a factory finally were authorized in 1937, and actual construction was begun in June, 1938. The first paper was turned out in September, 1939.

A major problem at Paramonga in the early days was the mastery of pulping techniques to produce the wide range of paper and paperboard qualities desired. Through trial and error a very crude pulp was found to be best suited to the manufacture of strawboard and corrugating medium—the fluted middle layer in corrugated paper—while in the production of sulphite wrapping grades a well-cooked unbleached pulp gave best results. At the start as many as six to eight different grades of pulp were produced, but in latter years the mill has standardized on four distinct pulp qualities.

Recently Paramonga has begun the production of bleached pulp on a small scale. At present the pulp is used in the production of colored sulphite wrappings, newsprint, and printing papers, but future plans call for the use of fully bleached bagasse pulp to produce other grades including toilet tissue.

Sugar cane bagasse is made up of relatively short fibers that will not withstand much beating or refining without undue fiber cutting and excessive hydration. Paramonga engineers, whose job it has been to select refining equipment and set up manufacturing procedures, have been guided accordingly. Degrees and methods of refining have had to be weighed against paper characteristics to assure economical production of satisfactory qualities at maximum speeds.

Paper made from bagasse, regarded somewhat skeptically when first introduced, has guaranteed its own acceptance by standing up well in actual performance. Bagasse newsprint is stronger than newsprint made from wood pulp. The advantages of paper sacks over cloth are beginning to be realized in export-import circles. A trial shipment of sugar from Peru to the United States in multiwall paper bags manufactured at Paramonga has strongly indicated the desirability of substituting this mode of packing for the cotton bags formerly used. The shipper's surveyor, rating the paper bags superior on several counts, reported that the new packing would "eliminate commercial loss resulting from dirty cargo...that contamination by foreign substance prevalent through single cotton bags is minimized through paper, and that the over-all strength of multi-ply paper is greater than cotton bagging."

Crush-resistance tests of corrugated boxboard made from bagasse proved Paramonga's product superior to wood pulp, and corrugated containers employing a bagasse medium were shown to be more crush resistant than corrugating materials normally used. The Quaker Container Co. of Los Angeles, Calif., which has purchased and used several hundred tons of bagasse corrugating medium declared it, "the most highly satisfactory paper our company has ever handled." In tests conducted by this firm combined board made with bagasse corrugating medium had a much higher crush resistance than competitive boards.

With its process thoroughly proved by the acid test of commercial production for a decade, W. R. Grace & Co. turned in 1950 to Sandy Hill Iron & Brass Works of Hudson Falls, N. Y., specialists in the manufacture of paper machinery for 93 years, to make its process available to the rest of the world. The Sandy Hill organization and W. R. Grace & Co. entered into an arrangement whereby the former was authorized to manufacture the machinery needed for the Paramonga process and to license the system to other cane growers and paper manufacturers.

Sandy Hill, a foundry established in 1858 in the heart of Upstate New York, moved into the manufacture of pulp and paper machinery as that industry grew. After gaining a solid reputation in the domestic field Sandy Hill broadened its scope of operations to include orders from Europe as well as from Latin America.

Widespread interest in the bagasse process, heightened by uncertain supplies of paper products in many parts of the world, has led to many inquiries concerning the W. R. Grace & Co. process, persuading the two companies to discuss their method before the public at this time.

OBITUARIES

Louvian Gardelle Simons

Louvian G. Simons, Paper Mill Engineer with Chas. T. Main, Inc., Boston, Mass, died on June 10.

Mr. Simons was born on Dec. 2, 1909, at Wisconsin Rapids, Wis. He graduated in chemistry in 1930 from the University of Chicago and received his master's degree in chemical engineering in 1932 from the Massachusetts Institute of Technology.

From 1932 to 1935 he was a report writer for V. D. Simons, Inc., in Chicago; 1935–37 plant engineer for the Flambeau Paper Co., Park Falls, Wis.; 1937–38 production engineer for the U. S. Gypsum Co. in Chicago; 1938–45 assistant chief engineer for the St. Regis Paper Co., Deferiet, N. Y. Since 1945 he was associated with Chas. T. Main, Inc., on pulp and paper mill engineering projects.

Mr. Simons was a member of the Technical Association of the Pulp and Paper Industry and a registered professional engineer in the State of New York. He is survived by his wife and two children.

John C. F. Darrah

John Clark Flagg Darrah, Chemical Engineer, Penobscot Chemical Fibre Co., Great Works, Me., died on Jan. 12, 1952.

Mr. Darrah was born in Richmond, Me., on Feb. 5, 1897, and graduated from the University of Maine in 1920 as a chemical engineer. He was employed by the Penobscot Chemical Fibre Co. since 1920.

TAPPI DIVISIONS AND COMMITTEES

Reports of Activities

Semichemical Pulping Committee

In December, 1951, F. W. O'Neil, Chairman, TAPPI Mechanical Pulping Committee, requested J. M. McGovern to act as chairman of a semichemical pulping subcommittee and to solicit members for this subcommittee. The following membership was established prior to the Annual Meeting of TAPPI in New York, Feb., 1952:

J. N. McGovern, Forest Products Laboratory, Chairman
C. W. Converse, Sprout-Waldron & Co., Inc., Secretary
Gerald Haywood, West Virginia Pulp & Paper Co.
R. G. Knechtges, The American Pulp & Paper Co.
H. J. Lausman, Consolidated Water Power & Paper Co.
J. F. Sapp, Gaylord Container Corp.
J. A. Staidl, Camp Manufacturing Co.
J. F. Stevenson, The Mead Corp.
C. K. Textor, The Bauer Brothers Co.

An organizational meeting of the above subcommittee was planned at the TAPPI Annual Meeting to convene at 4 p.m., February 18, following the committee meetings of the Pulp Manufacture Division. Because of the great interest in semichemical pulping, the meeting of the subcommittee was not actually held and instead a general discussion was held of the place of semichemical pulping activities within the organization of the Pulp Manufacture Division. Approximately 35 members of pulping committees and other interested parties were present for the discussion headed by K. O. Elderkin, General Chairman, Pulp Manufacture Division, TAPPI, and R. G. Macdonald, Secretary-Treasurer, TAPPI. It was decided at this discussion that the semichemical pulping activities should have full committee status in the organization of the Pulp Manufacture Division. Because of the large number of participants in the discussion and because of several strong opinions as to the exact scope of semichemical pulping, it was not possible to establish clearly the precise functions of the semichemical pulping committee. It was decided to adjourn the discussion to another organizational meeting to be held on February 21.

Organizational Meeting

The organizational meeting was called to order at 11 a.m. on February 21, in Room E, Hotel Commodore, New York, under the chairmanship of J. N. McGovern. The following members of the original subcommittee were present: C. W. Converse, R. G. Knechtges, J. E. Sapp, and C. K. Textor. J. S. Hart, Pulp and Paper Research Institute of Canada, was also present and accepted membership on the committee. R. G. Macdonald was likewise present to guide in the organizing of the committee. The following matters came up for discussion:

(a) It was agreed that the *scope* of activities of the semichemical pulping committee would include only those processes falling within the restrictions imposed by use of (1) wood chips as the fibrous raw material, (2) two-stage pulping procedures involving, first, partial chemical treatment of the chips, and, second, mechanical disintegration of the softened chips, and (3) pulps for paper and paperboard. It was understood that the so-called coarse fiber industry (wallboard and absorbent felts), which falls under certain definitions of semichemical pulping, fell elsewhere in the TAPPI organization. It was further understood that the so-called high-yield kraft and sulphite pulping would not be included in the scope of activities of the semichemical pulping committee.

(b) Mr. Macdonald recommended that all of the mills manufacturing semichemical pulp have representatives on the committee. It was agreed that this would be followed as much as possible. It was understood that the original members of the subcommittee would serve on the present committee.

(c) There was brief discussion of the number of meetings to be held of the committee. This point could not be settled until the actual activities were decided on but close cooperation was to be maintained with TAPPI section meetings and other pulping committee conferences.

(d) It was felt that the activities of the committee could best be conducted by subcommittees or task forces. A good model for this kind of organization is the Structural Fibrous Materials Committee, Industrial Division.

(e) The following activities were agreed on as suitable for starting the work of the semichemical pulping committee:

(1) Mill equipment survey.
(2) Standard terms.
(3) Index of degree of pulping.
(4) Cooking liquor test methods.
(5) Bibliography and abstracts.
(6) Periodic news letters.

J. N. McGovern, *Chairman*
C. W. Converse, *Secretary*

Chemical Products Committee

Better testing methods for tall oil and products containing tall oil was the principal subject of a general discussion which substituted for a formal meeting of the Chemical Products Committee.

There is particular need for a quantitative method for the rosin acids in tall oil or rosin especially where the rosin acids are present in only small amounts in products such as resins, paints, and other surface coatings.

The American Society for Testing Materials has two committees active in this work, Committees D-1 on Paint, Varnish, Lacquer, and Related Products and D-17 on Naval Stores.

Work is progressing toward a reliable quantitative method for rosin acids in various products accurate for amounts of rosin acids even below one per cent.

Such a method would outmode the present unsatisfactory Liebermaun-Storch Test. The present test has the defects that it is only qualitative and that it responds even to negligible amounts of rosin acids and, sometimes, to substances which contain no rosin acids.

Twenty laboratories recently cooperated in a trial of a new method for rosin acids, in tall oils or fatty acids containing rosin. The results with this method, developed by Richard Herrlinger and Gerald Compeau of the Arizona Chemical Co., were demonstrated to be very satisfactory.

For rosin acids contents below 15% the method appears preferable to the present Wolff method of ASTM Standard D-803.

Using six specimens ranging from 0.00 to 14.8% tall oil rosin acids, 85% of 114 determinations checked within 0.2 of the true value, 67% within 0.1.

Another group in ASTM is working to adapt these new methods to finished products like paints, and the evidence is already strong that this can be done.

Since there was no formal meeting there were no papers or reports on pending projects.

There is a growing interest in the chemical products of the pulp industry and readers of these brief notes are urged to join the Chemical Products Committee and help build a program of activities of benefit to the industry.

ARTHUR POLLAK, *Chairman*

Fundamental Research Committee

On May 14 the second meeting of the recently reorganized Fundamental Research Committee was held at The Institute of Paper Chemistry. Attending the meeting were Gerald Haywood, West Virginia Pulp and Paper Co., Rudy Griesheimer, The Mead Corp., J. R. Salvesen, Marathon Corp., D. T. Jackson, Hammermill Paper Co., W. M. Hearon, Crown Zellerbach Corp., J. C. Wollwage, Kimberly-Clark Corp., G. K. Dickerman, Consolidated Water Power and Paper Co., R. M. Dorland, Abitibi Power & Paper Co., and J. H. Fisher, Ontario Paper Co. and Chairman of the Canadian Fundamental Research Committee, together with Harry F. Lewis, Institute of Paper Chemistry, Chairman. The other member of the committee, G. A. Day of the Brown Co., was unable to attend the meeting.

Three major items of business were considered by the committee:

(1) It was decided that the committee would sponsor a symposium on management of research for the pulp and paper industry for the February TAPPI meeting. Plans are being made for full coverage of the subject and one full day's sessions will be organized. Among the possible subjects which will be considered are the following: notes and note taking, reports and communications; patent procedures; morale; research laboratory construction and design; stimulation of research; attendance at technical meetings; from the research laboratory to the development laboratory to mill procedures; recruiting of research personnel; publication policies; screening of research projects; what to do about reports; liaison with other research organizations and the internal organization of a research department.

The above classifications were made tentatively, and further organization of the program will no doubt change them considerably.

(2) Considerable time was spent in discussing the time, place, and program for the 1953 joint CPPA-TAPPI fundamental research conference on "Fundamentals of Paper Machine Operation." It was decided to hold this meeting preferably the week of September 14 in Appleton at The Institute of Paper Chemistry. It is hoped that a new auditorium will be constructed by that time and will be available for use for this meeting. The new facilities at the Institute will provide a very satisfactory location for this meeting. It was further decided that the symposium would include the fundamentals of sheet formation and removal of water by pressure—that is, papermaking up to the drier section. It was further decided that only the fundamentals of the processes will be discussed. No attempt will be made to consider the actual operations, for these fall within the perogative of other TAPPI committees. Among the general subjects suggested are the following: flow properties of pulp suspensions; flocculation; wire phenomena; new techniques and tools of use in such studies; and the mechanism and effect of wet pressing. It is planned to get outstanding speakers on these various subjects both from North America and abroad.

(3) The third general field discussed by the committee had to do with projects of potential interest to the Fundamental Research Committee. Some 50 or so projects had been suggested earlier by committee members; a number were selected from the list as having high priority, and soon these will be organized in the form of specific projects for submission to various research organizations by the Fundamental Research Committee. By this method the Fundamental Research Committee hopes to sponsor a group of significant projects in various institutions.

HARRY F. LEWIS, *Chairman*

TAPPI-ASTM Technical Committee on Petroleum Wax

FUNCTIONING AS TECHNICAL COMMITTEE M OF ASTM COMMITTEE D-2

Section I—Strength Tests

In order to present a clear picture of the current progress of Section I, it is necessary to give a brief résumé of past work of this committee as follows:

At the time Section I was originally formed, a comprehensive program was launched to study the precision of the various techniques in use at that time for determining the mechanical strength of paraffin waxes. These included both pure tensile and bending tests. This step was primarily a weeding out procedure.

As a result, certain methods were shown to give superior precision and from these it was established that the following fundamentals should be used in formulating a standard tensile strength test procedure.

1. A constant and uniform load applied to the specimen through suitable self-aligning specimen holders free of bending and torsion effects.
2. A standard rate of load application—namely, 80 pounds per second per square inch of specimen cross-sectional area.
3. Standard temperature and humidity conditions of test—namely, TAPPI Standards of 73°F. and 50% relative humidity.
4. A standard mold design for the preparation of test specimens of a streamlined type such as the Mellon Institute or modified dow mold.
5. Improve on methods of specimen preparation.
6. Scheduled periodic calibration of equipment with standard control samples.

Using the above fundamentals as a basis, it was recommended to the committee that a tentative test method be drafted incorporating certain modifications in specimen preparation. Using this tentative standard, a second round-robin program was initiated during the summer of 1951 with the main object of studying improved streamlined mold designs on an interlaboratory basis. Also, it was desired to study the modifications in specimen preparation.

Although a few of the participating members were not able to report their results in time for the meeting of Section I on Monday, Feb. 18, 1952, sufficient data were available to present a preliminary summary to the committee members with a complete report to be submitted at a later date. With the exception of the results on the Mellon Institute mold, the data still remaining to be reported should not change the over-all results or conclusions.

Therefore, based on the results of this comprehensive round-robin test program, it can be concluded that the improved mold designs studied will give superior precision based on statistical analysis and that, with the exception of certain minor changes, the tentative method of test as used in this program will not only give adequate but superior repeatability. With the exception of certain basic differences in calibration indicated in a few tensile machines, the test method should give adequate reproducibility.

The foregoing applies only to a test procedure for tensile strength. It was recommended by the chairman and agreed on by committee members that Section I should be divided into two subsections; one pertaining to tensile strength, the other to bending tests. Subsection chairmen will be appointed in the near future.

Both of these subsections will continue to function as a part of Section I with the object of arriving at acceptable

standard methods of test at the earliest possible date and to continue to improve on these methods when accepted.

The progress of this section was thoroughly discussed with the Advisory Committee and as a result it was their recommendation that this method be published in ASTM and TAPPI for information only in its present form, with the provision that final adoption of the mold design be made pending the decision of Section I committee members at a later date.

J. W. PADGETT, *Chairman*

Section II—Flow Tests

The results of the cooperative work on the modified penetration method, run according to the detailed instructions furnished to the participating members, were discussed. There was general agreement that the precision of the method as well as the agreement between the participating laboratories was satisfactory. Consequently, a motion by Mr. Padgett to publish "for information" the modified penetration procedure was carried. As stated in the motion, the modified penetration procedure is recommended at the present time only for use at 77 °F. and for waxes whose penetration does not exceed 50.

Since, however, there is considerable interest, especially in the paper industry, in information on the penetration of waxes at temperatures higher than 77 °F., it was decided to undertake cooperative work on the use of the modified procedure for determining penetrations at temperatures between 77 and 120 °F. A method of thermostating the specimens during the tests will be furnished to the cooperating laboratories and will follow the procedure used at present by Marathon Corp. and The Sinclair Research Laboratories, Inc. The samples to be used will be similar to those of the previous cooperative tests and the suppliers of the previous samples have agreed to furnish again the necessary samples.

In addition to using the modified test procedure at 77, 90, 100, 110, and 120 °F., tests with a special penetration needle will be carried out at these same temperatures. The purpose of this series of tests is to study the effect of the needle taper on the precision of the method. Standard Oil Development Co. will furnish a description of a special needle designed by them and will possibly furnish needles.

Because of the fairly widespread use of the Abrams consistometer for determining the hardness of waxes in the paper industry, consistometer readings on the cooperative test samples at the various temperatures used in the penetration tests will be obtained by those participating laboratories who have consistometers.

H. SCHINDLER, *Chairman*

Section III—Oxidation Stability

After discussing the results of the cooperative tests on standard samples, the committee agreed to continue work on the accelerated oxidation test while at the same time looking into methods which involved the direct absorption of oxygen. This test is carried out by maintaining the wax in a molten condition and bubbling air through it at a constant rate. The peroxide number is determined periodically and the curve of peroxide number vs. time plotted for the wax under test. It was decided to maintain the test temperature at 275 °F. for the present, with the understanding that for special cases other temperatures can be used. In order to approach more closely the point at which an oxidized odor first develops in a wax it was decided to measure the time required for a wax to develop a peroxide number of either 5 or 10 instead of the 50 previously used. The air rate was increased to 600 ml./min. (using a 75-gm. wax sample) to exceed the minimum required for oxidation rate to be independent of air flow (cf. *Petroleum Refiner* 22: 410–414 (1943)). The committee also agreed to investigate the use of O_2 instead of air. The O_2 rate was to be 150 ml./min. during this test.

There were two methods mentioned which measured extent of oxidation by the volume of O_2 absorbed, the Barcroft-Warberg method and the Shaker method. Mr. Yezek of General Foods volunteered to determine the stability of the four wax samples from the correlation test by the Barcroft-Warberg method and to forward part of these same samples to Mr. Hanson of California Research for determination of their stabilities by the Shaker methods. Results of these tests will be distributed (either by the chairman or directly) to the committee members for their consideration.

The following action is to be taken on the first-mentioned method. A copy of the method, revised to include all points discussed, will be distributed. Each committee member is to try out this method on available waxes of good and bad stability, determining if possible the repeatability, etc., of the procedure, and will forward test results and comments to the chairman as soon as possible. For each wax tested the time required to develop a peroxide number of 5 and 10 will be measured, and those members who are so inclined should also determine the time to reach a peroxide number of 50.

Any member of the committee who can do so should also investigate the use of O_2 as the blowing gas in place of air. A rate of 150 ml./min. O_2 should be substituted for 600 ml./min. air. (Use of O_2 may involve some fire hazard and ample precautions should be taken.)

As soon as data are available, the chairman will circulate a summary report on the method together with comments received to each committee member. A second cooperative test will then be initiated, the waxes to be used being selected from those tested during this preliminary investigation. Four to six samples, representing a wide variety of petroleum sources, will be involved.

G. P. HINDS, *Chairman*

Section IV—Sensory Tests

The purpose of the initial phase of the committee program was to obtain agreement on the following points:

1. Nature of the odor.
   (a) Satisfactory.
   (b) Unsatisfactory.
2. Test temperature.
   (a) Room temperature.
   (b) Elevated temperature.

Satisfactory odors were classified as (1) odorless and (2) sweetly paraffinic. Unsatisfactory odors were classified as (1) odors foreign to wax, such as solvent, lube oil, mercaptan or sulphides, musty, burned, etc., and (2) rancid odors, which may also be designated as an oxidized odor, but without necessarily being accompanied by a measurable peroxide value.

Seven samples of waxes typified by the above types of odor were obtained from two sources and submitted to the panel for testing on the basis of the following four types of odor tests.

1. Scrape a block of wax and smell the fresh shavings immediately while the shavings are in contact with the block of wax.
2. Melt the wax and pour a small amount on strips of odorless cellophane, allow to cool to room temperature, scrape the surface, and smell the fresh shavings while in contact with the solid wax.
3. Scrape shavings from the solid wax into a low-form, wide-mouth glass weighing bottle. Test immediately.
4. Place sample of wax to be tested in a low-form, wide-mouth glass weighing bottle, and raise to desired temperature and test immediately. Test temperatures were 140 and 180 °F.

The conclusions that can be drawn as a result of the initial work are as follows:

1. The various test panels could, in general, distinguish unsatisfactory waxes, particularly rancid waxes.
2. Of the waxes studied, the test panels indicated that there was better agreement using waxes from source A than from source B.

3. With wax from source A, the various test panels were able to select both satisfactory and unsatisfactory odors with an 80% or better incidence, irrespective of method.
4. With waxes from source B, the data indicate there was less general agreement between the various panels in the selection of satisfactory and unsatisfactory odors. In general, only two test methods showed a 60% incidence or better. These were methods 1 and 4—140°F.
5. The consumer member test panels find that the test methods had very little influence on odor evaluations.
6. The producer member test panels in general agree on only one method, namely, method 4, at 140°F.

As a result of this preliminary round-robin testing, it was decided to continue the work as follows:

1. *Samples and Tests.* Samples of waxes having a variety of odors, both satisfactory and unsatisfactory, will be obtained from five sources, and complete sets of samples will be submitted to each member of the test panel. Adequate precautions will be taken to prevent deterioration of the samples during handling and preparing for test.
2. *Evaluation.* The samples are to be tested at room temperature, 70–80°F., and at 140°F., as outlined in methods 2 and 4.

G. D. Fronmuller, *Chairman*

Section V-A—Composition

A meeting of Section V-A—Composition was held Monday, Feb. 4, 1952, at the Shoreham Hotel, Washington, D. C. The following points summarize the main actions taken.

1 The chairman discussed the status of the current test program in which we are endeavoring to establish a correlation between "oil" content values on microcrystalline wax, using in one instance methyl ethyl ketone at −25°F., and in the other ethylene dichloride at 0°F. There are seven different laboratories cooperating in a test program which involves the testing, in triplicate, of eight different samples of waxes of the aforementioned type. The work program is outlined in a letter from the chairman, dated Jan. 24, 1952. During the course of the meeting, the importance of each laboratory running exactly three determinations—no more, no less—in the interest of the statistical angle was stressed.

2. The Section voted in favor of republishing for information the Proposed Method of Test for Oil Content of Petroleum Waxes which appears as Appendix IV of the 1951 Report of ASTM Committee D-2 (see page 714 of the ASTM Standards on Petroleum Products and Lubricants).

3. In discussing the present precision section of D 721–51 T, Tentative Method of Test for Oil Content of Paraffin Wax, it was agreed that the chairman would review previous cooperative data, from the standpoint of some of the newer statistical concepts for reporting repeatability and reproducibility ranges. He will then report back to the Section as to whether there appears to be any need to change the precision section. In the latter connection, it is important to understand that no doubt is being expressed with respect to the precision of D 721–51 T.

4. The Section passed a motion recommending that Method D 721–47, Standard Method of Test for Oil Content of Paraffin Wax be dropped. For the past several years D 721–51 T has been published with a note to the effect that it is intended to replace D 721–47. Since D 721–51 T has been found satisfactory, the Section feels that the time has come to drop D 721–47.

J. C. Rather, *Chairman*

Section VI—Blocking

The chairman opened the meeting by asking Mr. Farnham, of the Standard Oil Development Co., to give a description of their temperature gradient method for determining the blocking point of paraffin waxes. This method consists essentially of placing machine-coated strips of wax paper in contact with each other under a weighted bar on a metal plate while maintaining a temperature gradient across the metal plate by means of baths. Mr. Farnham showed data on the number of waxes which indicated reproducibility within 2°F. The temperature gradient used is approximately 3°F./in. A hand-coater has been constructed and is used satisfactorily for preparing the test specimens.

Mr. Mueller, of the Marathon Corp., reported on a similar method which also appears to give excellent reproducibility and correlates quite well with service performance of waxed sheets.

After considerable discussion the committee agreed that since the mercury method, which had been investigated previously, has one very serious disadvantage of possible mercury poisoning hazard, the temperature gradient method should be investigated. The following procedure was agreed on. A complete description of the two temperature-gradient procedures will be submitted to the chairman, who will distribute them to the committee. Two 1-lb. samples of paraffin wax will be distributed to each member of the committee by seven suppliers. It was agreed that the wax load on the test sheets would be set at 5 to 10 lb. surface wax per side per ream (3000 sq. ft. ream). The test will be run for 17 hr., and no more than two specimens will be used in each stack. A 1 by 1-in. steel bar will be used as the pressure medium.

The question was raised as to whether more work should be done in correlating penetration with blocking. It was the consensus of the committee that our efforts should be confined to a blocking test and that correlation with penetration, a test under study by the Flow Properties Section, is not a function of the committee at present.

M. W. Kane, *Chairman*
A. C. Smith, Jr., *Secretary*

Section VII—Sealing and Laminating

The progress of cooperative testing as set up at the Dec. 8, 1950, New York meeting was summarized in a typed sheet passed out by Mr. Borden.

The program as set up consisted of two groups of tests—one on a waxed sulphite sheet, and the other on a microwax laminated cellophane. The laminated cellophane part of the program was not carried out because it was impossible to get the desired amount of wax on the cellophane sheet with the available laboratory equipment. The seal strength tests with the waxed sulphite sheet were carried out as planned.

The test data of the cooperating laboratories indicated the necessity of standard conditions as follows:

1. Preparation of the sample.
2. Constant period of aging before testing seal.
3. Rate of delamination.
4. Angle of delamination.
5. Conditions of temperature and humidity.

The laboratories of Moore & Munger, Socony-Vacuum Oil Co., and Riegel Paper Corp. cooperated in a limited test program planned at a get-together of some of the committee members at the ASTM meeting on June 21, 1951, in Atlantic City. This work was carried out with glassine paper and a commercial laminating agent. The test results in general confirmed the above general conclusions but, in addition, indicated the amount of wax or laminant must be also controlled in order to obtain reproducible results.

After considerable discussion it was agreed that a test method be drawn up based on the following conditions and published in *Tappi* some time this year for information and comment.

Paper for Sealing Strength Tests. George Mueller, of the Marathon Corp., will provide a supply of a paper to be used as a standard. It will be a 25-lb. sulphite sheet meeting the following requirements:

Basis weight (24 × 36—500)	24.5 to 25.5
Mullen test at 95 ± 10 ml./min. glycerine flow	14 p.s.i.

The DRAPER Felt
DRAPER BROTHERS COMPANY
CANTON, MASS.

Tear	
With grain	20 min. gm./16 sheets
Cross grain	20 min. gm./16 sheets
Moisture	5 to 7%
Density	13 to 15

The paper will be conditioned at standard TAPPI conditions (73°F.—50% R. H) at least one week before coating for seal strength testing.

Coating Any method of coating will be used so long as the finished sheet, saturated with wax, shall have 3½ lb. ± ½ lb. R surface wax on the sealing side (unfilled side). The waxed strip shall be a minimum of 5 in. wide in order that a 3-in. test specimen can be prepared by cutting 1 in. from each side.

Sealing. The Palo-Myers method, using air cooling at room temperature (70 t) 80°F.), will be used to seal the waxed sheets together. The sheets are to be sealed back to back (unfilled side t) unfilled side) and in the machine direction of the paper. The test specimen will be a 3-in. strip cut from the center of the sheet (a minimum of 1 in. to be cut from each side) so that it can be peeled parallel to the length of the original sheet.

Constant Period of Aging Before Testing Seal. The sealed specimens will be conditioned by storing at standard TAPPI (73°F.—50% R. H.) for 24 hr. before peeling.

Rate of Delamination. The rate of delamination will be 2½ in. per minute, which is equivalent to a separation rate of 5 in. per minute of the two ends.

Angle of Delamination. The two sheets shall be separated in the same plane at an angle of 180°F. so that the unseparated portion shall be at right angles to this plane.

Instrument for Separation of Sheets. Any instrument that will measure the grams force necessary to separate the two plys and has a sensitivity of 1% of the minimum load to be applied can be used to measure the bond strength of the sealed or laminated sheets. Examples of such instruments are: basis weight scales, jolly balance, tensile testers, etc.

Reporting Results. The results will be reported as grams force per centimeter width; also the report will include the actual width of the specimen used in the delaminating test.

The angle of delamination was discussed and the consensus was the 180° angle appeared to be the simplest, most commonly used, and would show the same relative results as tests using a different angle.

Discussion regarding the procedure for sheets laminated with microwaxes indicated that the same general procedure could be used except the test specimen would be 1 in. in width and glassine would be used instead of the sulphite sheet. The results will be reported in grams force per centimeter width.

Another round-robin of tests using the proposed procedure was agreed upon. The committee members who received samples for the program set up at the Dec. 8, 1950, meeting will be contacted by the secretary to find out if the samples are available and also to see if they desire to participate in th cooperative test program. Mr. Borden will furnish a roll glassine paper to each cooperator for the lamination streng test.

GEORGE C. BORDEN, JR., *Chairma*
J. H. KIRK, *Secretary*

Section VIII—Scuff and Gloss

Results of round-robin tests performed during the year we discussed with reference to the chairman's report to the Se tion dated Feb. 6, 1952. Because of the more or less negati results of the tests, it was decided to do more fundament work to develop better instrumental methods, since prese instruments are inadequate.

Scuff. More work will be done, using the Marathon scu tester, in searching for a satisfactory scuffing surface. Su faces to be tried include abrasive glass, a fine steel file, a dry standard paper. In addition, a different principle scuffing will be investigated using an instrument design su mitted by the Shell Oil Co. To correlate test results wi commercial equipment, waxes with scuffing characteristi observed on converting and/or packaging machinery will included in this work.

Gloss. Dr. MacLaren, of Indiana, presented the results his work on gloss using a Hunter goniophotometer and a visu comparator which employs some of the principles of the Hunt instrument. Since his results were most promising, it w agreed that he would continue those studies and advise th Section chairman periodically on his progress. Since M Hunter is cognizant of the work going on, it was also decide to ask him for any assistance he might be in a position contribute.

In the past, The Institute of Paper Chemistry has studi the problem of the measurement of gloss of papers. In ord to take advantage of their experience, it was suggested th they be contacted to see what they might contribute to th particular problem.

GEORGE P. MUELLER, *Chairman*
A. P. ANDERSON, *Secretary*
TAPPI-ASTM *Technical Committ on Petroleum Wax*

THIRD TAPPI TESTING CONFERENCE

State University of New York College of Forestry

Syracuse, N. Y., Sept. 10–12, 1952

International Standards

International Testing Procedures

The following report was presented at the International Conference held at Appleton, Wis., on Sept. 17, 1951, by R. G. Macdonald, Chairman of the Subcommittee on Testing and Nomenclature, Wood Chemistry Committee, United Nations Food and Agriculture Organization.

It was prepared in cooperation with the chairmen of the testing committees of the technical organizations similar to TAPPI in Australia, Canada, Germany, Great Britain, and Sweden.

Its publication has been delayed in order to obtain and include in the report the testing procedures having official status in the above-mentioned societies.

The report follows:

On July 5, Dr. Harry F. Lewis of The Institute of Paper Chemistry wrote to the chairman as follows:

"In connection with the F.A.O. meeting to be held in Appleton in September, do you have a list of the TAPPI standard methods which are also accepted as standards in Great Britain, in the Scandinavian countries, and in Australia? There is a desire to start a standardization program and it was my thought that there were some methods which are essentially the same in the various associations and which might be set up for approval as F.A.O. standards."

The chairman responded to Dr. Lewis as follows:

"I have your letter of July 5 in regard to the possibility of establishing some international testing methods.

"The possibility of doing so at present is limited, I believe, to methods that do not involve the use of physical testing devices that are not universally used. Most chemical methods should be possibilities for international standardization.

"Following is a statement by D. I. Sutherland, Secretary of the Australian Paper Makers Testing Committee, published in the April, 1951, news letter of APPITA. This will illustrate the problem insofar as pulp testing is concerned":

The pulp and paper industry uses a wide variety of testing methods on raw materials, on finished products, and during production.

Before the formation of the A.P.M. Testing Committee, each of the laboratories in A.P.M. obtained its own set of testing instructions for those tests in which it was interested independently of the other laboratories.

In most cases the laboratory took the testing instruction from TAPPI or the British Paper Makers' Association. As these instructions had been prepared after critical examination of the method, they were accepted without question. Unfortunately it was found that the TAPPI instructions gave insufficient detail, with the result that different operators were unintentionally making different interpretations of the instructions, and as a result were using different procedures. This was first evidenced with the water resistance test, where differences between results obtained by different observers were found to be due to this reason. A further matter which cast a shadow on the TAPPI methods was the tearing resistance method, which gave an instrument specification which was not met by any of the instruments in general use in Australia, and, as far as is known, elsewhere.

To eliminate these faults a testing committee was set up under the chairmanship of the chief works chemist, and consisted of the chief chemist at each mill, the chief chemist of central laboratory, and the research laboratory superintendent.

One of the first tasks undertaken was to set up an index of testing methods to include those in use at that time, and to allow for future methods which may be desired. The index was classified into five main sections: preparations, equipment, mill operating, testing, and miscellaneous.

Listed in this index were over 200 methods, and the committee then tackled the task of preparing drafts of these methods. As a first step regulations were prepared covering the procedure to be used in writing standard methods, and the methods were divided into groups which were allocated one to each mill.

Since that time the mills have been working through the lists and, as a result, 40 standard methods have been prepared. Attention has in general been concentrated on methods where differences have been found, or where there is no recognized method already available. It should be mentioned that for most of the remaining 160 methods the TAPPI methods have been used by all operators without encountering the above-mentioned faults.

This correspondence was forwarded to Roger C. Griffin, Chairman of the TAPPI Standards Committee and to James d'A. Clark, General Chairman of the TAPPI Testing Division.

Mr. Griffin responded:

"I am in substantial agreement with you regarding the use of TAPPI methods for international standards. I believe that any TAPPI Official or Tentative Standard test methods not dependent upon test apparatus which is not readily available outside of the United States might be suitable as a basis for international standards. I would not include the TAPPI Suggested Methods."

Dr. Clark responded:

"There are a number of TAPPI methods that could well be considered for this purpose (international standards). Among them would be T 11 m, T 201 m, T 17 m, T 220 m, and possibly T 205 m, T 410 m (restricted to grams per square meter), T 411 m, and possibly T 404 m.

"The main difficulty with pulp and paper testing is the difficulty in obtaining agreement as to standard conditioning atmosphere. I have been urging the British and Australians to accept 50% R. H., but they prefer to use 65% R. H. This is unfortunate because the tests are empirical anyway and what could be lost from using testing conditions a little drier than their average would be made up by the great gains to be derived as a result of the common understanding that would accrue if all results were shown on the same basis of relative humidity."

A letter was sent to the testing committee chairmen of the associations in Australia, Canada, Great Britain, Sweden, France, and Germany reading as follows:

"I am enclosing herewith an Index to the TAPPI Standards. If your association has a similar list I would appreciate it if you will send me a copy.

"On Sept. 17, 1951, there will be a meeting of the Food and Agriculture Organization of the United Nations at Appleton, Wis. The writer is acting as the coordinator of testing procedures for the Wood Chemistry Committee.

"At this meeting some consideration will be given to the possibility of establishing international testing methods. To this end will you please indicate if any of the methods of your association are substantially the same as certain TAPPI Standards. It is quite likely that some of the chemical methods of testing are very similar.

"I would appreciate it if you will send me a brief report of the work of your testing committee since 1948.

"If you care to do so please let me have your ideas concerning the possibility of establishing international standards and indicate, in particular, which of your methods fit this possibility."

The following responses were received:

Commonwealth Scientific and Industrial Research Organization Division of Forest Products, Box 18, Post Office, South Melbourne S. C. 4, Victoria, Australia

Dear Mr. Macdonald:

"In reply to your letter of August 6, only a few methods have been considered as possible Australian methods. Where existing methods are found to be satisfactory it is not proposed to issue Australian standards. However, where investigation has shown existing methods to be unsatisfactory, or where the special conditions of the Australian industry make it necessary, standard methods will be issued by the Australian Pulp and Paper Industry Technical Association (APPITA), probably in collaboration with the Standards Association of Australia.

"The methods which have been considered to date are: the measurement of internal tearing resistance; the determination of permanganate numbers of pulps; the sampling of pulp for moisture content. The first-mentioned method is the only one which has been completed by the Testing Committee, the other two being still under investigation.

"The method for the measurement of internal tearing resistance differs from the TAPPI method in that four sheets of paper are torn together in all tests, irrespective of the weight of paper or of the reading on the testor. Sectors which are respectively twice as sensitive and half as sensitive as is the TAPPI Standard sector are used for weak or extra strong papers. The APPITA method also specifies a tear testing instrument which actually conforms with the TAPPI specification, and we believe it to be the only one which does this, unless the Thwing-Albert Instrument Co. has recently altered the design of its instrument. This method, the draft of which has been approved by the Testing Committee, will be sent up to the Standards Committee of the association for its consideration in the near future.

"The question of international standards has been discussed by the APPITA Testing Committee, and there is general agreement on the desirability of having standards which are common throughout the world. As mentioned above it is not proposed to prepare and issue APPITA standard methods when existing methods are satisfactory. It is very desirable to have an organization to which suggested standard methods could be submitted for discussion and comment before acceptance as international standards. Such a procedure would go a long way toward the setting up of the latter. However I believe that this is a function of the International Standard Organization and not of the F.A.O. Technical Committee on Wood Chemistry. As was suggested at the Brussels meeting in 1949 the F.A.O. Committee's function would be to provide the background research which must of necessity precede the drafting of the standard method."

W. E. COHEN, *Officer-in-Charge*
Wood Chemistry Section

Technical Section of the British Paper and Board Makers' Assoc. (Inc.)
St. Winifred's Welcomes Road, Kenley, Surrey

Dear Mr. Macdonald:

"Your letter of August 6 addressed to Mr. Prior has been forwarded to me as I am now dealing with that side of the Paper Testing Committee's liaison activities which Mr. Prior formerly handled.

"As I am sure you must be aware, the paper testing methods published by the Technical Section up to 1947 were: The First Report of the Paper Testing Committee and Standard Method for the Determination of the Thickness and Bulk of Paper. At that time the First Report went out of print and the committee decided to revise the individual methods but to publish such revisions as separate booklets. The work of revision was put in hand, and up to date two of these have been published, namely:

Standard Method for the Determination of the Thickness and Bulk of Paper (PT 5, sm: 1950)

"In 1948 four new testing methods were put forward:

Testing the Strength of Wet Paper (PT 14, pm: 1947)
Testing the Sizing Properties of Paper (PT 15, pm: 1947)
Absorbency Test for Bibulous Paper (PT 16, pm: 1947)
Rapid Methods for Determining Whether Waste Paper (Especially Wet Strengthened Waste) Can Be Readily Disintegrated for Re-use (PT 17, pm: 1948)

"The first three of these were presented at the Fifty-Third General Conference on March 19, 1947. Subsequently, two further methods were presented at the Fifty-Eighth General Conference on Oct. 12, 1949:

Sampling and Testing Paper for Moisture Content (PT 18, pm: 1949)
Determination of the Permeability to Water Vapor of She Materials (PT 19, pm: 1949)

"The committee has also recently published two furthe new methods:

Determination of the Ash Content of Paper (PT 20, pn 1950)
The Detection and Estimation of Nitrogenous Treating Agen in Paper (PT 21, rp: 1951)

"I enclose the full range of our methods which also include below the names of those now available. Revision of th outstanding methods of the First Report is proceeding an the printing of PT 2, 3, and 4 is well in hand. During th coming nine months the committee hopes to complete som of the remaining eight methods.

"The Paper Testing Committee is not really concerne with wood chemistry or chemical tests: this would com within the terms of reference of our Pulp Evaluation Com mittee. The matter has indeed been considered in anothe connection and it was found that there is no significant con tribution available.

"The Paper Testing Committee is very anxious that ther shall be international accord so far as this is possible in pape testing and the methods specified and, for this purpose, th arrangement instituted under Mr. Prior's aegis was brough into being. The scope is, of course, limited to yourselves, th Canadian Technical Section, and APPITA. Perhaps th establishment of international standards is a much more am bitious project and while my Paper Testing Committee, I am sure, subscribes in principle to its desirability, I think tha difficulties of varying degrees of intractability are almos bound to arise when details come to be considered. My Pape Testing Committee is due to meet on October 5 and I shall c course place your letter before it then. If, as a result, ther is anything to add to my present letter, I will write to yo again.

"Following is detailed a complete list of the British PBM standard methods for testing papers, which have been pub lished to date by the Paper Testing Committee.

PT 1: sm 1951 Standard Method for the Determination o the Thickness and Bulk of Paper
PT 2: rp 1951 Sampling Paper for Testing
PT 3: sm 1951 Conditioning of Paper Test Samples
PT 4: sm 1951 Paper Substance (Ream Weight) Determ nation
PT 5: sm 1950 Folding Test
PT 6: sm 1937 Tearing Test
PT 7: sm 1937 Bursting Test
PT 8: sm 1937 Bursting Test
PT 9: sm 1937 Bursting Test
PT 10: sm 1937 Air Permeability Test (T. Trevor Pott method)
PT 11: sm 1937 Air Permeability Test (Gurley Densomete
PT 12: sm 1937 Tensile Test
PT 13: rp 1937 Notes on the Application of Statistics t Paper Testing
PT 14: pm 1947 Testing the Strength of Wet Paper
PT 15: pm 1947 Testing the Sizing Properties of Paper
PT 16: pm 1947 Absorbency Test for Bibulous Paper
PT 17: pm 1948 Rapid Methods for Determining Whethe Waste Paper (Especially Wet Strengthened Waste) Can B Readily Disintegrated for Re-use
PT 18: pm 1949 Sampling and Testing Paper for Moistu Content
PT 19: pm 1949 Determination of the Permeability t Water Vapour of Sheet Materials
PT 20: pm 1950 Determination of the Ash Content o Paper
PT 21: rp 1951 The Detection and Estimation of Nitro enous Treating Agents in Paper

(Methods PT 2: rp 1938 to PT 13: rp 1937, inclusive, a the methods which originally appeared as The First Report of th Paper Testing Committee. This is now out of print and th methods are being revised before reprinting—e.g., PT 5—Th Folding Test.)

"In assigning a distinctive number to this and each of th other published methods for testing paper the committee ha employed the following system:

Paper testing methods are indicated by the prefix PT

"The following descriptions are used abbreviated in the method number as shown:

rp—recommended procedure
sm—standard method
pm—provisional method

"The year of publication or subsequent modification is shown after the method number."

FRANCIS BOLAM, *Secretary*

Institut für Cellulosechemie
Darmstadt, Germany

Dear Mr. Macdonald:

"Your letter of Aug. 6, 1951, has apparently crossed with my letter of Aug. 10, 1951.

"Enclosed please find a list of the standard methods of our association. When comparing this list with the TAPPI Standard Methods, there seems to be only a certain similarity concerning our Merkblatt 5, 6, and 8. Besides, as I wrote you already, we are especially concerned about our new alpha-cellulose-standard IV/29 of which I sent you some copies. I believe that this method could be the basis for an international method, since it includes the two possibilities, that means working with and without dilution.

"As Chairman of the Fiber Analysis Committee I may report to you, that since 1948 we have been chiefly occupied with establishing the new alpha-cellulose method. Furthermore we are contemplating to edit a method for the rapid determination of copper viscosity of pulp according to Dr. Doering. We are also working on standardizing the procedure of moisture determination in dry and wet pulp. Further methods under consideration are swelling ability according to Jayme, lignin determination modified, resin determination in pulps, and others."

Following is a list of the *Standards* of the *Verein der Zellstoff- und Papier-Chemiker und -Ingenieure* (Verein Zellcheming) (Association of Pulp and Paper Chemists and Engineers):

PULP PRODUCTION COMMITTEE

Standard I/1 Determination of Specific Gravity r_0 and Moisture Content of Wood
Standard 302 Standard Method for Testing of Lime Stones

PAPER PRODUCTION COMMITTEE

Standard II/1 Specific Gravity Tables of Aluminum Sulphate Solutions

FIBROUS MATERIALS TESTING COMMITTEE

Standard 1 Characterization of Pulps. Characterization of Unbleached Pulps Regarding Pulping Degree (Hardness) and Bleachability
Standard 2 Determination of Pulping Degree (Hardness) of Unbleached Sulphite and Soda Pulps
Standard 5 Determination of Resin and Fat Content of Pulps
Standard 6 Standard Methods for Determination of Alcohol Extract and the Isolation of Unsaponifiable Matter, Fatty Acids, and Resin Acids from Resin Extracts
Standard 8 Standard Method for Determination of Copper Number of Pulps
Standard 9 (A) Determination of Wood Gum in Pulps. (B) Determination of Pentosans ("Tollens Number")
Standard 10 Standard Method for Determination of Swelling Criteria
Standard 11 Standard Method for Determination of Xanthate Viscosity
Standard 13 Standard Method for Testing Pulps Regarding Their Mechanical Parchmentizing Ability
Standard 14 Standard Methods for Testing Sulphite Waste Liquors
Standard 15 Microscopical Testing of Pulps Regarding Rosin
Standard 16 Standard Methods for Testing Sulphite Waste Liquors (Supplement to Standard 14)
Standard 17 Qualitative Determination of Lignosulphonic Acid (Second Supplement to Standard 14)
Standard 18 Standard Methods for Testing Pulps Regarding Color Stability
Standard 20 The Testing of Writing Papers by Means of Drawing Pen Lines
Standard IV/23 Standardized Dyestuffs for the Analysis of Fibrous Materials
Standard IV/24 Substitution of Iodine by Chloramine ("Chlorina") for Analysis
Standard IV/25 Supplement to the Assortment of Standardized Dyestuffs for the Analysis of Fibrous Materials (Supplement to Standard IV/23) with Table I: Supplement to the Assortment of Standardized Dyestuffs for the Analysis of Fibrous Materials and Table II: Assortment of Standardized Dyestuffs for the Analysis of Fibrous Materials
Standard 26 Physical Constants of Sulphite Waste Liquor (Supplement to Standards 14 and 16)
Standard 27 Determination of the Absorption Power of Pulp Wadding
Standard 28 Determination of the Thickness Swelling Volume of Pulp Sheets (Supplement to Standard 10)
Standard IV/29 Determination of Alpha-Cellulose and Alkali Insoluble Fraction of Pulps

PULP STRENGTH TESTING COMMITTEE

Standard 101 Standard Method for Testing Strength of Pulps. (A) Historical Survey
Standard V/2 Standard Method for Testing Strength of Pulps. (B) Theoretical Basis
Standard 103 Standard Method for Testing Strength of Pulps. (C) Storage of Pulp and Its Preparation for Strength Testing
Standard 104 Standard Method for Testing Strength of Pulps. (D) Defibering of Pulp for "Nonbeaten" Test
Standard VI/05 Standard Method for Testing Strength of Pulps. (E) Beating of Pulp
Standard 106 Standard Method for Testing Strength of Pulps. (F) Equalization of Beaten Pulp and Its Distribution
Standard 107 Standard Method for Testing Strength of Pulps. (G) Determination of Slowness (Beating Degree)
Standard 108 Standard Method for Testing Strength of Pulps. (H) Forming of Handsheets
Standard 109 Standard Method for Testing Strength of Pulps. (J) Conditioning of Handsheets
Standard 110 Standard Method for Testing Strength of Pulps. (K) Partitioning and Cutting of Handsheets.
Standard 111 Standard Method for Testing Strength of Pulps. (L) Testing the Conditioned Pulp Sheets for Weight/m², Thickness, Apparent Density, Moisture Content
Standard 112 Standard Method for Testing Strength of Pulps. (M) Testing the Conditioned Handsheets for Breaking Length, Burst Factor, Folding Endurance, Tearing Resistance
Standard 113 Standard Method for Testing Strength of Pulps. Evaluation of Results

MECHANICAL PULP PRODUCTION COMMITTEE

Standard 201 Suggested Method for a German Standard Method for Testing Quality of Mechanical Pulps
Standard 202 Suggested Method for the Classification of Mechanical Pulps

Swedish Paper and Pulp Engineers Association
Stockholm, Sweden

Dear Mr. Macdonald:

"In answer to your letter of August 6 where you request some notes on work performed by the Swedish Association Analysis Committee, I am sending you herewith some information of this kind. These notes have been put together by the Chairman of our Committee Miss Karin Wilson of Skoghall.

"Herewith also is a list of testing methods published by the Swedish Association."

RAGNAR SODERQUIST

Work done since 1948 and planned future work 1951—since 1948 the Swedish Analysis Committee has been working on the following methods:

Determination of Dry Substance in Sulphite Waste Liquor Method CCA 22.
Determination by Evaporation of the Residue in Black Liquor Method CCA 23
Determination of the Pentosan Content of Rayon Pulps Method CCA 24

(This method is similar to method CCA 4 up to and including the first distillation of the furfural, which then is colorimetric determined with orcin. The results check quite well with the results of the old method CCA 4, but the determination is less time consuming and gives more reliable results for pulps with very low pentosan content.)

The Swelling of Pulp Sheets During Mercerizing

A method is adopted but not yet published. It is, however, the same method which is recommended by Ringstrom and Apler (*Svensk Papperstidn.* 51: 501–508 (1948)).

Forming Handsheets for Brightness Test of Pulp

Some work has been done on this subject and it has been found that the result is very dependent on the design and material of the apparatus for sheet forming. Distilled water must be used. The work is not yet finished but we feel that it would be of great interest to have a standard method for preparing samples for brightness testing, especially as the result is very dependent on the method used.

Determination of Viscosity of Pulp in Cupriethylenediamine

Since 1948 the main work of the committee has been concerned with a method for viscosity determination. Part of this work has been summarized in a report which was sent to Milton Fillius and Peter Van Wyck on Sept. 14, 1949. Since then we have done some work on the viscosity measurement and the design of the viscometer with special consideration of the importance of reporting the viscosity at a specified velocity gradient. A new method for the dissolving of the pulp has also been worked out. The pulp is first slurried in a stable neutral solution of copper chloride-ethylenediamine. After addition of 1.4-*N* NaOH the pulp is dissolved. This method is very simple and gives results with good reproducibility. The standard deviation for analyses in 6 different laboratories was ±1–2% for rayon grade pulp and ±3% for high-viscosity pulp. We intend to publish this method this year as a tentative standard method and we hope to receive comments and criticism on it from foreign committees before adopting any new viscosity method as a standard method.

Planned new work for the near future:

Alpha-Cellulose and Alkali Solubility

The TAPPI method T 203 m, the German "Merkblatt" method No. 7, and the Swedish method CCA 7 are founded on the same principle, but differ in several details which are of great importance for the result. The three methods consequently do not give the same result. As the alpha-cellulose value is of great importance when selling and buying pulp we consider that it would be of great value to have an international standard method. We feel, however, that a determination of the alkali solubility in 18 and 10% NaOH without subsequent washing with water would characterize the pulp better. This alkali solubility can be determined volumetrically according to CCA 8. Through G. Jayme we have quite recently received a new German method for gravimetric determination of alkali solubility in 18 and 10% NaOH (Merkblatt IV/29 Verein der Zellstoff und Papier-Chemiker und Ingenieure). Dr. Jayme has asked us to comment on this method. We, therefore, plan to compare this and some other methods with our own method CCA 8 and to start some work on how to determine and report the alkali solubility properties of pulp most adequately.

Determination of Cooking Degree of Pulp

Recently, two articles on this subject have been published in the Finnish Journal *Papper och Tra.* R. Gustafson (*Papper och Tra* 2, 5B, 6B Nos. 4A, (1950)) has written a literature review over methods for determination of cooking degree and P. Alander (*Papper och Tra* 33, No. 6: 201 (1951)), has determined the correlation between several methods. As some of these correlations do not check with the correlations published in CCA 9 we plan to investigate whether our curves in CCA 9 must be changed. We feel that an international standard or at least internationally adopted curves for recalculating the results of the different methods to a standard method would be of great value.

Those methods which in our opinion would best fit in an international standard have been marked with X in the list of our methods.

We consider it to be of particular interest to get international standard methods for determination of viscosity and alkali solubility (alpha-cellulose).

CCA no.	*Accepted*	*Title*	*Corresponding[a] TAPPI method*	*Suitable international standard*
1	1940	Determination of Ash in Pulp	T 211 m	. .
2	1940	Determination of Resins and Fats in Pulp	. . .	X
3	1940	Determination of Copper Number	T 215 m	X
4	1940	Determination of Pentosans	T 223 m	X
5	1940	Determination of Lignin in Wood	T 222 m	X
		Lignin in Unbleached Pulp	T 13 m	. .
6	1940	Determination of Wood Gum		
7	1940 (rev. 1946)	Determination of Alpha-Cellulose	T 203 m	X
8	1941	Determination of the Alkali Solubility of Cellulose According to Billerud	. . .	X
9	1941	Determination of the Cooking Degree of Pulp	T 202 m T 214 m	X
10	1941	Determination of Beta- and Gamma-Cellulose	. . .	X
11	1942	Determination of Reducing and Fermentable Sugar in Sulphite Waste Liquor According to Modified Scheerl Method	. . .	. .
12	1942	Determination of Alcohol Yield from Sulphite Waste Liquor by Fermentation	. . .	. .
13	1942	Determination of Cuprammonium Viscosity According to TAPPI[b]	T 206 m	. .
14	1942	Methods of Analysis for Sulphate Turpentine	. . .	. .
15	1945	Analysis of Crude and Distilled Tall Oil[b]	. . .	. .
16	1944	Quick Method for the Determination of Cuprammonium Viscosity	. . .	X
17	1944	Determination of Strength Properties of Chemical Pulp After Beating in Lampén Mill	T 205 m T 220 m	. .
18	1944	Analysis of White Liquor	T 624 m	. .
19	1944	Determination of Ash and Ash Components in Rayon Pulp	. . .	X
20	1947	Determination of Sulphate in Sulphite Waste Liquor	. . .	. .
21	1947	Determination of the Mean Gage of Pulp Sheets	. .	. .
22	1948	Determination of the Dry Substance in Sulphite Waste Liquor	. . .	. .
23	1948	Determination of Evaporation of the Residue in Black Liquor	. . .	. .
24	1950	Determination of the Pentosan Content of Rayon Pulps	. . .	X
25	1950	Swelling of Pulp Sheets During Mercerizing	. . .	X

[a] The corresponding TAPPI method is in principle but not exactly the same. Only CCA 3, TAPPI 215 m, and CCA 13, TAPPI 206 m are identical.

[b] Summary.

The testing methods are distributed by Svensak Papper och Cellulosaingeniorsforeningen, S. Blasieholmhamnen 4 A, Stockholm, Sweden.

KARIN WILSON, *Chairman*
SPC *Fiber Analysis Committee*

Technical Section, Canadian Pulp & Paper Association
Montreal, P. Q.

Dear Mr. Macdonald:

"I have assembled some material in reply to your letter of August 6.

"These include an Index to the Technical Section Standards with notes relating to present work and a list of CPPA Standards that differ from the TAPPI Standards. In the list of CPPA Standards the corresponding TAPPI Standard code number is given.

A. MEASURING AND SAMPLING

Basic Density of Wood	A-1
Weighing, Sampling, and Testing Wood Pulp for moisture.	A-2 (T 210 m)
Sampling Paper and Paperboard for Testing	A-3 (T 400 m)
Conditioning Paper or Paperboard for Testing	A-4 (T 402 m)
Sampling of Chemical Pulps in the Pulp Mill for chemical Testing	A-5

Flow Measurements of White Waters and Wastes	A-6 (E 2 p)
B. Identification	
Fiber Composition of Paper	B-1 (T 401 m)
C. Fiber Treatment	
The Determination of Freeness	C-1 (T 272 m)
Laboratory Processing of Pulp (Beater Method)	C-2 (T 200 m)
Laboratory Processing of Pulp (Ball-Mill Method)	C-3 (T 224 sm)
Forming Handsheets for Physical Tests of Pulp	C-4 (T 205 m)
Forming Handsheets for Optical Tests of Pulp	C-5 (T 218 m)
D. Physical Testing	
Machine Direction of Paper and Paperboard	D-1 (T 409 m)
Wire and Felt Sides of Paper and Paperboard	D-2 (T 455 sm)
Basis Weight of Paper	D-3 (T 410 m)
Thickness and Density of Paper and Paperboard	D-4 (T 411 m)
Bulking Thickness of Paper or Paperboard	D-5 (T 426 m)
Tensile Breaking Strength of Paper and Paperboard	D-6 (T 404 m)
Stretch of Paper	D-7 (T 457 m)
Tearing Resistance of Paper	D-9 (T 414 m)
Wax Test for Surface Strength of Paper	D-11 (T 459 m)
Physical Testing of Pulp Handsheets	D-12 (T 220 m)
Sieve Analysis of Pulpwood Chips	D-13
F. Absorptive Properties	
Time of Water Penetration into Paper and Paperboard (Dry Indicator Method)	F-1 (T 433 m)
Water Absorption of the Surface of Paper and Paperboard ("Cobb Test")	F-2 (T 441 m)
Surface Wettability of Paper and Paperboard (Angle of Contact Method)	F-3 (T 458 m)
Absorption by Bibulous Papers of Water and Writing Ink	F-4 (T 432 m)
Oil Resistance of the Surface of Paperboard	F-5
G. Chemical Testing	
Moisture in Wood Chips and Sawdust (Toluene Method)	G-1 (T 3 m)
Moisture in Pulp by Toluene Method	G-2 (T 208 m)
Moisture in Paper and Paperboard	G-3 (T 412 m)
Water Solubility of Wood	G-4 (T 1 m)
Water Solubility of Pulp	G-5 (T 207 m)
One Per Cent Caustic Soda Solubility of Wood	G-6 (T 4 m)
One Per Cent Caustic Soda Solubility of Pulp	G-7 (T 212 m)
Lignin in Wood	G-8 (T 13 m)
Lignin in Wood Pulp	G-9 (T 222 m)
Ash of Pulp	G-10 (T 211 m)
Ash of Paper and Paperboard	G-11 (T 413 m)
Pentosans in Pulp	G-12 (T 223 m)
Pitch in Wood Pulp	G-13 (T 204 m)
Resin in Paper	G-14 (T 408 m)
Paraffin in Paper and Paperboard	G-15 (T 405 m)
Chlorine Consumption of Pulp	G-16 (T 202 m)
Permanganate Number of Pulp	G-17 (T 214 m)
Amount of Mineral Coating on Paper and Paperboard	G-19 (T 407 m)
Alcohol-Benzene Solubility of Wood	G-20 (T 6 m)
J. Nonfibrous Material Testing	
Analysis of Bisulphite Cooking Liquor	J-1
Analysis of Bleaching Powder, Bleach Liquor, and Bleach Sludge	J-2 (T 511 m)
Analysis of Sulphur-Burner Gas	J-3 (T 603 m)
Analysis of Limestone	J-4 (T 618 m)
Analysis of Lime	J-5 (T 617 m)
Analysis of Alum	J-6 (T 614 m)
Sampling and Analysis of Coal	J-7 (T 605 m)
Analysis of Mineral Fillers	J-8 (T 615 m)
Indicators for Volumetric Analysis	J-9 (T 609 m)
Analysis of Bisulphite Cooking Liquor	J-1
Analysis of Bleaching Powder, Bleach Liquor, and Bleach Sludge	J-2 (T 611 m)
Analysis of Sulphur-Burner Gas	J-3 (T 603 m)
Analysis of Limestone	J-4 (T 618 m)
Analysis of Lime	J-5 (T 617 m)
Analysis of Alum	J-6 (T 614 m)
Sampling and Analysis of Coal	J-7 (T 615 m)
Analysis of Mineral Fillers	J-8 (T 615 m)
Indicators for Volumetric Analysis	J-9 (T 609 m)
Analysis of Rosin Size	J-10 (T 628 m)
K. Terminology	
Standard Terms for the Furnish of Paper and Paperboard and for the Measurement of Fiber Losses	K-1
Standard Terms Used in the Alkaline Pulping Process	K-2 (O 400 p)
Z. General	
Rules for the Preparation and Adoption of Standard Methods	Z-1

Standards to Be Issued Shortly

D-13 Sieve Analysis of Pulpwood Chips—Now Standard
J-1 Analysis of Bisulphite Cooking Liquor—Revised Standard
J-2 Analysis of Bleaching Powder, Bleach Liquor, and Bleach Sludge—Revised Standard

Physical and Chemical Standards Committee March, 1951

1. Species Identification of Wood and Wood Fibers
2. Bisulphite Liquor Analysis
3. Bleach Liquor Analysis
4. Evaluation of Clay
5. Analysis of Soda Ash
6. Analysis of Lime
7. Identification of Specks and Spots in Paper
8. Preparation of Volumetric Solutions for Analysis
9. The Mullen Test (Institute Project)
10. Useful Methods

In connection with Item 10, "Useful Methods," a request was sent to Branch Chairmen in 1948 for routine control methods in use in mills, which might be of interest to other mills. Several replies were received, and it is proposed to edit and publish these for comment, with the end in view of gradually building up a collection of useful mill methods. This would also serve the purpose of "trying out" potential standard methods for several years before recommending them as standards.

Technical Section, CPPA Standards Which Differ from Corresponding TAPPI Standards

A6 (E 2 p) Flow Measurements of White Waters and Wastes. A6 omits data on submerged weirs.
C2 (T 200 m) Laboratory Processing of Pulp (Beater Method). Considerable differences.
D3 (T 410 m) Basis Weight of Paper. D3 omits paperboard.
D6 (T 404 m) Tensile Breaking Strength of Paper and Paperboard. D6 has different specification for test specimen.
F4 (T 432 m) Absorption by Bibulous Papers of Water and Writing Ink. Considerable differences.
J1 (T 604 m) Analysis of Bisulphite Cooking Liquor. J1 being revised to include gravimetric method. Present standard omits it.
J5 (T 617 m) Analysis of Lime. J5 being revised. Method for determining available lime will be changed.
J10 (T 628 m) Analysis of Rosin Size. J10 includes an additional method.
K2 (O 400 p) Standard Terms Used in the Alkaline Pulping Process. K2 has some differences in definitions.

Tappi Standards

The list of Standards of the Technical Association of the Pulp and Paper Industry is published in its Year Book. (A copy of the list will be sent to individuals needing it to supplement this report.)

The following items relate to changes that have been made since 1949:

Two new Tentative Standards were approved during the year and submitted to the Secretary for publication:

T 19 m-50 Pentosans in Wood
T 1000 m-50 Thermal Conductivity of Structural Insulating Board

A revision of Official Standard T 9 m-45, Holocellulose in Wood, was approved and published and will later be submitted to letter ballot of the TAPPI membership for ratification.

A revision of Tentative Standard T 12 m-47, Specific Gravity (Density) and Moisture Content of Pulpwood, was approved and published.

Two Tentative Standards submitted to letter ballot of the TAPPI membership in the Spring were advanced to Official Standards:

T 475 m-47 Bleeding Resistance of Asphalted Papers at Elevated Temperatures

T 632 m-47 Preparation of Magnesium Oxide Standard for Spectral Reflectance

It was decided to delay advancement of three others on the letter ballot, because of adverse criticisms.

Tentative Standard T 221 m-42 was approved, in corrected form, for advancement to Official Standard. It will be published and then submitted by letter ballot for ratification.

Two Suggested Methods were advanced to Tentative Standards:

T 800 sm-44 Drum Test for Fiberboard Shipping Containers

T 805 sm-45 Water Resistance of Shipping Containers (Shower Method)

At year's end four submissions were under consideration by the Standards Committee:

Forming Structural-Type Boards for Physical Tests of Pulp (new)

Specular Gloss of Paper at 75° (new)

Contrast Gloss of Paper at 57.5° (revision of T 424 m-45)

Specifications for Chrome-Nickel-Iron Alloy Castings for Sulphite Pulp Processing Equipment (revision of E 400 s-43)

Note: The report of the TAPPI Standards Committee for 1951 appears in *Tappi*, 35, No. 2: 81 A–84 A (Feb., 1952). The information supplements that given in this report.

Discussion Comment

This report was discussed at length at the meeting of the Wood Chemistry Committee on Sept. 16–18, 1951.

There were several objections presented to the proposal that international standards be adopted through the medium of the F.A.O. It was felt that there are now international standardizing agencies in existence and that the F.A.O. for the present should limit its activities to being a clearing house wherein the standardizing activities of the separate pulp and paper technical organizations may make known to each other their individual progress in developing testing procedures.

R. G. Macdonald, *Chairman*

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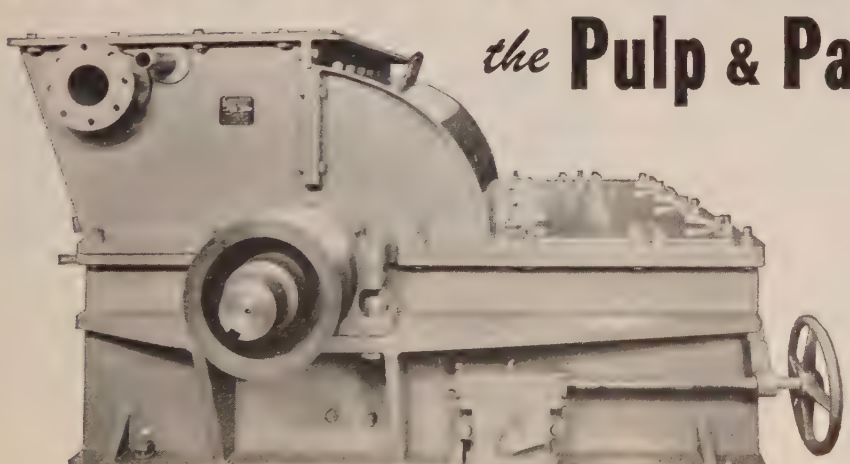

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LOCAL SECTION ACTIVITIES

Reports of Meetings, Personnel, and Events

Ohio

The Ohio Section completed its most successful year to date with a dinner meeting attended by over 225 persons on May 8 at the Hotel Manchester, Middletown, Ohio. Prior to the meeting Shartle Bros. Co. entertained the Section at a cocktail party.

The annual election of officers was held with the following men elected to serve for the coming year:

Chairman—C. E. Brandon, Howard Paper Mills, Inc.
Vice-Chairman—J. L. Clouse, Oxford-Miami Paper Co.
Treasurer—R. D. McCarron, Stein-Hall and Co.
Recording Secretary—G. B. Grigg, Cincinnati Gas and Electric Co.
Corresponding Secretary—Lee Buechler, Crystal Tissue Co.
Executive Committeemen—P. S. Cade, Harding-Jones Paper Co.; Arthur Thurn, Champion Paper & Fibre Co.; D. J. Goodman, Sorg Paper Co.; H. A. Smith, Mead Paper Co.; M. G. Lyon, Champion Paper & Fibre Co.

Entertainment for the evening was arranged by Fred Holt of Brown-Bridge Co. Radio, television, and night club entertainers, from Cincinnati, provided an enjoyable evening.

H. A. Smith, retiring chairman, presents gavel to C. E. Brandon, chairman elect

Retiring Chairman Herb Smith expressed his appreciation to his officers and to the Section members for their work and coöperation the past year. Three meetings were held in towns other than Middletown which has always been the meeting place for the Section. The total attendance for the year considerably exceeded that of any previous year. The incoming officers are already making plans for continuing the successful type of programs practiced this last year.

R. M. Morris, Jr., *Recording Secretary*

Executive Committee Meeting of May 27

The first meeting of the Executive Committee of the Ohio Section for the year 1952–53, was held Tuesday, May 27, 1952, at the Manchester Hotel in Middletown, Ohio, with Chairman C. Edwin Brandon presiding. The members present were: Herbert A. Smith, John W. Clouse, Wm. C. Martin, Arthur Thurn, Lee Buechler, Richard M. Morris, Harold Joiner, Mal Lyon, and George Gregg.

The retiring chairman, Herbert A. Smith, expressed his appreciation for the fine work done by the retiring Executive Committee and the other committees who worked with him.

Treasurer R. D. McCarron was unable to be present but through Chairman Ed Brandon he reported that we lost $175 on our 1951–52 operations even though the price per member attending meetings was increased from $2.75 to $3.50 beginning with the December, 1951, meeting at Battelle Memorial Institute in Columbus, Ohio.

The following appointments were made and approved by the committee:

Routine Control Methods Committee:
Harold Koch, *Chairman*, 2-yr. term, Weston Paper Co.
George H. Sheets, 2-yr. term, Mead Corp.
Clifton Towles, 1-yr. term, Champion Paper & Fibre Co.
E. K. Lum, 1-yr. term, Beckett Paper Co.
Chas. Hill, 3-yr. term, Sorg Paper Co.
C. E. Ahlm, 3-yr. term, Chillicothe Paper Co.

Data Sheet Committee:
H. P. Petzold, *Chairman*, 2-yr. term, Oxford-Miami Paper Co.
W. G. Printz, 2-yr. term, Manchester Machine Co.
J. Baxter, 1-yr. term, Shartle Bros. Machine Co.
Milton Jones, 1-yr. term, Mead Corp.
R. Bamman, 3-yr. term, Champion Paper & Fibre Co.
Harold G. Suhs, 3-yr. term, Sorg Paper Co.

Publicity Committee:
John W. Clouse, *Chairman*
Harold Joiner
Mal Lyon and the assistant editor he chooses to help him to edit the publication "Ohio Tappi"
Morgan Robinson, Photographer
G. B. Gregg

John L. Clouse moved that the publication "Ohio Tappi" be continued for the year 1952–53. H. A. Smith seconded this motion. The motion was passed. Coeditors Mal Lyon and Don Jenkins were congratulated on the fine work in getting "Ohio Tappi" started and Mal Lyon reported that he will have to find another coeditor for the 1952–53 season. The monthly cost of this publication is $40 to $45.

George B. Gregg, *Recording Secretary*

Empire State (Central District)

The May 2 meeting of the Empire State (Central District) Section was held at the University Club in Syracuse. There was a social hour from 6:00 until 7:30 and the dinner began immediately thereafter. Ninety-two attended.

This meeting was the annual Senior Dinner. Empire State TAPPI played host to the seniors and was represented by Ralph Prince, treasurer.

Left to right: Harry Gray, newly elected vice-chairman, C. E. Libby, present chairman, Vance Edwardes, speaker of the evening, Daniel Lent, present secretary-treasurer, W. R. Willets, present chairman, Metropolitan District, Empire Tappi, and Hilliard Cook, newly elected chairman, Central District, Empire Tappi

The nominating committee (George Martin, chairman, Fred Sommerville, Durwood Geffken, C. P. Donofrio, and George Nicholson) presented its nominees who were then voted on. The results of the election show that the following men will guide the Central District next year:

Hilliard D. Cook, *Chairman*
Harry Gray, *Vice-Chairman*
Earl Church, *Secretary*
Robert Hitchings, *Treasurer*

The seniors were addressed by Vance Edwardes, past-president of TAPPI. His words of wisdom were directed to help the young men entering the field of papermaking.

This meeting was, without doubt, the best meeting held all year.

DANIEL V. LENT, *Secretary*

Opportunities in the Paper Industry

Vance P. Edwardes

PAPER is a big business but it is doubtful if it is recognized as such by those who are not, in some way, directly or indirectly connected with its manufacture or sale. Paper is essential to our way of life and civilization but even the

June graduates of the Pulp and Paper Department of the State University of New York, College of Forestry. Standing: Left to right: Cruz, Fischer, Cerasoli, Schutts, Abelson, Miller, Crowell, Lorey, Sweitzer, Wally, Holmberg; Seated, l. to r.: Hyde, Rothe, Snyder, Prof. Libby, Hamilton, Knapp, Finnegan. Absent: Gladstone, Slentz, Kraiman

government failed to recognize that fact during the last war until almost too late. (About 30 tons of blueprint paper are required for the plans for one battleship.) Over the years, the production of paper has followed a straight line when plotted on semilog scale, i.e., 6% compounded semiannually, and the per capita consumption has kept pace until the production and consumption for 1951 have reached nearly 29 million tons and 382 lb. per year for every man, woman, and child in the United States.

It has been said that the per capita consumption of paper is a measure of a nation's achievement along the lines of economic, cultural, and educational advancement. While at present this may not be strictly true, it is certain that in the recognized backward countries the per capita consumption falls under 5 lb. per year. Even in the Western European countries it is still but a fraction of the United States figure. Great Britain is the highest with about 135 lb. while the European average is about half of that figure. Just try to visualize what would happen in this country if our paper consumption were cut in half—most of us might not even be here—there would be no comics, no 25¢ editions of classics or mystery novels, no individual food packaging, and newspapers and magazines half size.

As big as the industry is and as rapidly as it has grown, there have been relatively few outstanding changes in manufacture except mechanical ones—larger digesters, wider and faster machines—but basically there have been no fundamental changes. The industry is conservative and many companies have the attitude of "Let George do it." The amount spent for research, while gradually increasing, is still far below that invested by other chemical industries.

You so often hear that there are no more opportunities in industry—that everything has already been discovered. Don't you believe it. It is my feeling that the surface has only been scratched and that developments in the years to come will make the present state of the art seem like the dark ages.

It has always seemed downright silly to prepare two types of pulp, one containing practically all the original wood substance and the other a purified chemical product to get a mixture of the two. How much better it would be to prepare a product containing the desired proportions of the combination or having the required properties. Semichemical pulp is a step in that direction as is also the process of preliminary impregnation of wood prior to grinding which has been further developed at Syracuse.

Wood has been and is the mainstay of the industry but, after all, what we are dealing with is cellulosic materials and there are many besides wood available. True, up till now, annual crops could not compete on a cost or quality basis but now the gap is narrowing, if not already closed, on both counts. The problems of collection and storage have been pretty well solved and that of quality, definitely so. These other pulps are, however, best used as extenders for wood pulp. For the purpose of this discussion let us confine ourselves to wood.

Is there any reason why trees cannot be made to grow faster even in cold climates? Recent startling results have been secured in promoting more rapid growth of animals by the use of hormones. Does anybody know that research along similar lines will not promote forest growth? It can be done for garden plants but can it be done economically for the forest trees? It is true that a hybrid poplar with extremely rapid growth has been successfully developed with plantations both in this country and Europe. According to test results, yield and strength are excellent by any of the standard chemical pulping methods.

So far, nobody has found an economic use for bark so why can it not be removed in the woods, thus saving transportation costs, decay on storage, and wood loss in barking? The recently chemical debarking methods which have been under study for a few years, may be an answer to that question. Even the installation of a chipping plant at large woods operations might be feasible under proper conditions.

Of all the major industries, paper knows the least about its raw material. Wood is bought on a volume basis and the product sold on a weight basis. With but few exceptions, the pounds of cellulosic material purchased is an unknown quantity yet a change of 0.010 in specific gravity will change the yield of chemical pulp about 30 lb. per cord. Also, unbeaten sulphite pulp bursting strength will vary directly with gravity—that is true for softwoods and may be also true for hardwoods. The industry recognizes, to some extent, the effect of gravity on yield but so far, has been unable to do anything concrete about it, probably because of wood-buying policies.

Wood storage, or rather storage under unfavorable conditions, is the source of a large but unknown loss both in wood substance and pulp quality. Naturally many variables control the rate of decay; rough wood will decay more rapidly than peeled, wet more than dry, sanitation of the wood yard: piling methods, wood rotation, and species all have a bearing. Naturally, such studies as have been made show widely divergent results but it can be assumed that decay starts in 6 mo. after cutting at the rate of 0.5% a mo. for the first year and that rough wood, random piled, will suffer about a 40% loss at the end of 3 yr. Naturally you will wonder why something is not done to reduce this loss. There are probably several reasons: until recent years wood supply was plentiful and cost reasonable; very few mills have the means to measure such losses—weightometers and automatic samplers are not in common use, and other wood measurements are too crude

VANCE P. EDWARDES, Consultant, Corinth, N. Y.

to reveal the loss; the determination of pulp production or yield is generally a matter of guesswork and finally, insofar as book costs are concerned, it is cheaper to store the wood in random piles, which require less space than ricked piles, and to use wood as it is being delivered to the mill rather than piling it and using older wood from the pile for current operations.

Wood preparation, barking, cleaning, and chipping each take their toll of wood. Wood substance loss in barking will run from about 2% for a stream barker; 4% for a drum barker, and about 12% for a knife barker, all on small wood. Excessive cleaning can cause a 20% loss on individual sticks, and chipper sawdust, depending on the condition and operation of the chipper, accounts for 1 to 6% additional loss.

With wood, in the Northeast, costing about $40 a rough cord, delivered, the importance of wood conservation at every step of the process is obvious. To illustrate what can be done it is only necessary to recall the savings in wood and man power which resulted from the development of the hydraulic barker for western logs—a saving of over 20% in wood and 100 men in the wood room.

In a mill producing bleached pulp and equipped with a modern, preferably three-stage bleach plant, it is questionable if thorough cleaning of the wood before chipping is necessary. A study of this problem could result in a worthwhile wood saving.

The operation and care of the chipper is another step which will bear watching, both with respect to possible wood savings and improved pulp quality. With a well-maintained and operated chipper, sawdust can be kept down to 1% but let maintenance be slack and operation sloppy, sawdust can easily rise to 6%. Quite generally, under poor conditions, chip uniformity suffers with resultant high pulp screening loss, and pulp strength is lowered due to chip damage caused by excessive bending of one end of the chip which results in fiber fracture.

The preceding is common to all chemical pulping processes but from here on the discussion will be confined largely to sulphite.

There are always opportunities for more efficient acid plant operation—particularly important in view of the present shortage of sulphur together with the demand for increased production. Losses as great as 5% can easily occur under poor operating conditions. The causes are: poor burner operation with low or irregular gas concentration resulting in the formation of excessive amounts of SO_3 and sublimation. Leaky coolers, admitting either water or air, cause further SO_3 formation and poorly operated absorption systems add to the loss through gas vented to atmosphere. Automatic controls for burner sulphur level and gas concentration are available and their intelligent use will reduce sulphur losses to a minimum. Routine checking of the SO_2 concentration at the vent of the absorption system is important in keeping loss from this source low.

Acid storage tanks must be kept tight since they are, at times, under a slight vacuum. Air leaks, wherever they occur always result in SO_3 formation.

It is in the operation of the digesters, with respect to pulp yield and gas recovery, that the possibilities for large savings exist. The hot-acid system has made these savings easier to realize and, when combined with forced circulation, chip packing and, if economic conditions warrant, indirect heating, the results can be excellent provided intelligent use is made of the added equipment. However, poor cooking control and gas recovery can nullify the gains made possible by the added equipment. Satisfactory automatic temperature, pressure, time controls are available and, while expensive, are justified by increased pulp uniformity and better gas recovery.

The production of sulphite pulp has been practically at a standstill for several years but the agitation against stream pollution may be a blessing in disguise. It has forced the industry to very actively investigate the use and recovery of soluble bases, such as magnesium, sodium, and ammonia together with heat. While all the problems have not been solved on chemical recovery, remarkable progress has been made which, in the end, could reduce sulphite pulp costs, even in this country, as much as $5.00 a ton. The magnesium-base plant at the Weyerhaeuser Longview plant has been operating for several years and gradually making improvements in chemical recovery. A Norwegian mill has developed a feasible recovery method for soda-base liquors, but as yet no economic recovery method has been developed for ammonia. In Scandinavian and European countries, where economic conditions differ from those on this continent, it is a paying proposition to recover as little as 40% of a calcium-base waste liquor for the heat value alone. With approximately 100% recovery of the solids and irrespective of base, a very appreciable amount of SO_2 can be recovered from the boiler stack gas. With soluble bases the recovery is easier than with calcium base liquors but up to 150 lb. of sulphur per ton can be recovered in any case. Progress along these lines should be followed closely, and opportunities exist for worth-while developments.

Continuous pulping has been the dream of inventors for years but until recently no satisfactory equipment has been designed. The most common example is that which is finding increasingly wider use in the board industry, known as the Asplund and that manufactured by Pandia. After years of pilot-plant development, the Swedish firm of Kamyr, a research association of three manufacturing firms, has now in operation a continuous kraft digester and is actively working on the development of a continuous sulphite digester.

It is unfortunate that so few mills have any reliable figures on pulp yields or even pulp production. There is no other sure way by which the result of operational changes can be judged. Of course, a mill producing dried pulp does know production but not always yield. However, there are means available for determining both wood consumption and slush pulp production.

Appreciable losses can and do take place in the screening operation, irrespective of pulp quality. Most of these are caused by human failure resulting in irregular operation which, in turn, reduces screening capacity and cleanliness. Sometimes a contributing cause lies in the lack of adequate equipment but most can be laid to consistency and volume variations resulting from careless operation. The installation of a venturi weir will indicate the extent of screenings loss but until automatic consistency and volume controls are installed ahead of the screens, losses will always be higher than necessary and production and cleanliness poorer.

In the beater room opportunities exist for saving the more costly ingredients of the furnish by assuring proper amounts of each are used and not more added, often by the whim of the machine room but sometimes because of carelessness on the part of the beater man. There are many ways in which losses can occur and a study should be made covering all possibilities. One common occurrence is overfurnishing on an order. When that happens, the beater of stock is, more often than not, dumped into the sewer. The same can happen from errors in furnish, particularly color. The installation of a central furnish control system or individual flowmeter control of all items entering the furnish of each machine, while expensive, is justifiable.

Savealls of various types are practically standard equipment on all paper machines, some very efficient and some not so good. The determination of losses in white water is an important means of keeping losses from this source minimum, but the problem is not simple. Most often sewers are so located that it is next to impossible to properly sample and measure the flow but some means must be found to do so if excessive loss is to be avoided.

The foregoing has attempted to enumerate some of the operational problems which will be encountered in every self-

contained mill but now, at the risk of offering unasked advice, I would like to mention some of the personnel problems you will run across.

I have no figures as to how many graduates of pulp and paper courses stay with the industry but I imagine it will be higher than the 40% figure which used to apply to engineering graduates. There is something fascinating about the industry that, once in it, men seldom leave. Then there are the various pension plans, all designed to keep the employee working for the same company. However, it would be a mistake to continue working under distasteful conditions just to build up retirement pay.

While there is not as much changing from one company to another as in the past, it is not a bad policy to change jobs while you are still young, always provided there is a sound reason for doing so. Shifting around can be carried to extremes, and employers look askance at a man who has changed jobs too often unless the reasons are justifiable. When leaving one job for another, always leave in such a way that they would be glad to rehire you at some future date.

If you go into operation, remember that shift work may be necessary for some years and, unless an agreement has been reached between the company and the union, you will be faced with seniority rules. Generally speaking, pay is better, also chances for advancement, the closer, in the manufacturing line, an employee is to the customer.

Your starting salary is not an indication of your worth to the company—they probably will not break even on you for a year—it is only that they are short of technically trained men and have to pay that much to get you. After all, your success will depend roughly 20% on education and 80% on your ability to get along with men and put your ideas across.

Learn the relationship between departments, how each is dependent on all others. Be observant and prepare yourself for the job ahead. Unless you are enthusiastic about your work it will be far better for both you and the company to find a job which does appeal to you.

Never underestimate the practical operator who has had little formal education but who has spent years in the industry. Get on his right side and you will learn a lot; buck him and you are in for trouble but together you will be an unbeatable combination.

Chicago

At the May 19 meeting of Chicago Section, Alfred P. Haake, nationally known economist, consultant, and Mayor of Park Ridge, Ill., addressed the group on "What's Ahead for Business?" Mr. Haake spoke of the trend toward more and more government control and evaluated rather seriously the effect that government regulations have on the economy. He stated that our prosperity is not an altogether healthy prosperity, that purchasing power has been reduced 50%, currency has been inflated, and that savings have been destroyed.

V. V. Vallandigham, A. P. Haake, and Peter Trucano

Speakers' table of the May 19 meeting of the Chicago Section: J. R. Fulghum, Socony Vacuum Oil Co.; W. R. Price, Applied Research Service; V. V. Vallandigham, Kelco Co.; A. P. Haake; and Peter Trucano, Morris Paper Mills

He further stated that the new generation has adopted the feeling that the government will provide and that it does not have the zest for adventure that the older generations have had. The pioneer spirit is not present. These are not altogether healthy signs.

We have also increased our productivity enormously and now inventories in many lines are catching up and there may have to be some adjustment in prices, Mr. Haake said.

Our economy, Mr. Haake stated, is tied in, of course, with the world situation and he does not think the Russians are serious in wanting truce. Consequently, he looks for no letup of business on account of the relieved foreign situation.

WARREN R. PRICE, *Chairman,*
Publicity Committee

New England

A joint meeting of the Connecticut Valley Division of the Superintendents Association and the New England Section of TAPPI at Toy Town Tavern, Winchendon, Mass., May 16 and 17, drew one of the largest attendances ever recorded in the history of both organizations. More than 300 members and guests enjoyed the fine program of highly interesting papers and the sports and other recreational features.

Helen U. Kiely, American Writing Paper Corp., assisted by Paul Applegate, Hercules Powder Co., was in charge of the program and arrangements. Assistance also was given by John Retallick, Secretary of the Connecticut Valley Division, and C. C. Decker, Crane and Co., Inc., the golf chairman.

This meeting opened on Friday morning, May 16, with registration. Following luncheon, the first of the business sessions was held in the nearby Playhouse and featured the presentation of four excellent talks on "Functional Papers." Harold W. Knudson, Hollingsworth & Vose Co., Chairman of the New England TAPPI Section, presided and presented the speakers.

"Permanent Papers" was the topic discussed by Lewis S. Reid, Metropolitan Life Insurance Co., New York, N. Y. He stressed the importance of permanency in paper (100% rag) when his company selects paper for its applications, policies, and records—all three of which must stand up under usage up to 100 yr. and over. Mr. Reid outlined some of the more

They discussed functional papers at Winchendon, Mass., meeting, May 16. Left to right: Charles Woodcock, General Foods, Inc.; Philip L. Staats, General Electric Co.; Lewis S. Reid, Metropolitan Life Insurance Co.; and Harry O. George, International Business Machines Co.

important requirements in this type of 100% rag paper as: it must be made right, treated properly in the beaters, run slow on the machine, and conform to set size, strength, and opacity tests. In the specifications, he said, there must be accuracy, precision, workability, suitability, flexibility, and acceptability.

Mr. Reid said that in fairness to both there must be a good understanding between the buyer and the maker of paper in regard to specifications, and added that 100% ledger, index, and letterheads are also material for his company which have to be kept for many years, up to 100 yr. or more.

The speaker said that 75% rag paper and even 50% rag bond made today will last as long as 100% bond made 30 yr. ago and that there is an inclination due to high costs to downgrade from a set policy of 100% rag bond. He told of how papers in the files of his company for 50, 75, and 100 yr. have turned brown and where handled frequently are frayed on the edges and gone on the fold.

Harry O. George, of the Laboratory Division of International Business Machines Co., at Endicott, N. Y., discussed "Business Papers." He told of how his company goes about setting specifications for the many grades of paper used in the different types of business machines made by his company. Mr. George said that paper is of high importance in conjunction with the accurate operation of business machines and such papers include cards for punching, paper tape for machine mechanism control, bond paper forms, and answer sheets for use in Army and school tests.

Mr. George stated that specifications, with proper tolerances, for his company's use are based on functional satisfaction, with uniformity most important and specified thickness a vital machine function factor. He said that factors required in punched cards are proper basic weight, thickness, strength, durability; in paper tape, durability for maximum life; moisture content with its important bearing on the retention of flatness by the card or paper used, on registration of printing and retention of dimensions of the card. Mr. George said that every roll of paper used in his company is tested by an electrical method in regard to moisture content and faults are corrected with an Emerson drier. The speaker said that the coefficient of friction of the punch cards is important and that curl, conductivity, electrical properties, sulphur or sulphide, abrasives in the cards or paper are objectionable.

"Electrical Papers" were discussed by Philip L. Staats, head of the Paper Laboratory, General Electric Co., Pittsfield, Mass. Mr. Staats stated that paper and paper products for electrical insulation are one of the large problems of his company and he classified six different papers used in the electrical field—foil, slot, saturating, condenser, pressboard, and vulcanized.

Mr. Staats spoke of the objectionable items in papers used in his field and touched briefly on specifications. He named as objectionable such items as conductive particles, electrolytes, and ash and said that paper or board must have dielectric strength which is a combination of uniformity, strength, and specific gravity. Mr. Staats said that whatever can be done to reduce particles in the paper or paper products his field uses will furnish a big improvement in electrical papers. He said that in the manufacture of the most desirable paper for electrical insulation care must be used in the selection of the grade aad quality of the pulp and its treatment in preparation. In this regard he said that spruce pulp has proved the best thus far.

Charles Woodcock, of the Central Research Laboratory, General Foods, Inc., Hoboken, N. J., made "Packaging Papers in the Food Industry," the theme of his discussion. Mr. Woodcock said that the big factors in specifications for food packaging papers are moisture resistance, wetproofing and transparency. Speaking of protective factors he named moisture resistant paper, with waxed paper, as the grade generally used for packaging cereals, bread, crackers, etc. He named transparency as an important merchandising factor and said that in the final analysis it is a matter of what is best in the finished food packaging which is the ultimate test of what best meets the many requirements involved.

Mr. Woodcock spoke of the use in his industry of wax laminated sheets, glassine, glued bags, aluminum foil, plastic foil, foil paper waxmounted, greaseproof and wax laminated glassine. He dealt with the qualities required in parchment for the packaging of frozen foods to keep out any possible oil from printing imprints; of packaging material tests with shortening, coconut oil and wax; of the great problem in properly packaging coffee which, when ground, gives off gas and thus makes it difficult to have a package that will liberate the gas yet hold out damaging moisture. He said that in designing the coffee package there must be a precise measurement of the vacuum required.

Mr. Woodcock said that he would like to see developed a film of paper that will retain its characteristics from and during winter and tropical temperatures. He then named other difficulties in securing the ideal packaging; such as, completely greaseproof carton board that will withstand high sterilization temperatures; an easy opening and easy closing package that will withstand heat and cold and moisture.

H. W. Knudson (left) greets Alfred Montagna, chairman of the Connecticut Valley Division of the Superintendents' Association

Following the presentation of these four papers there was a brief question period.

After dinner, John B. Calkin, head of the Pulp and Paper Foundation, University of Maine, held attention and interest with a brief talk on the necessity for industry to aid in the training of students for technical positions in pulp and paper mills. Mr. Calkin spoke of the dependence of the technical men on the superintendent and reviewed briefly the growth of the technical man in the pulp and paper industry since 1941. He said that the past 25 yr. have brought papermaking to a precise science and that more and more the technically trained man now figures in the practical operation of pulp and paper mills.

Mr. Calkin spoke of the efforts now well under way to train young men for careers in pulp and papermaking and revealed that since 1912, when the first pulp and paper courses in the country were started at the University of Maine, more than 600 of the Maine alumni have come into our industry.

Mr. Calkin commended the institution at the University of Maine of P. S. Bolton's plan for a 5-yr. course in managerial training in pulp and paper mill operations and Fred Soderberg's initiation of the plan to establish the Pulp and Paper Foundation at the University of Maine recently.

The speaker outlined the Foundation's plan of instruction to students and this plan has received widespread support from many mills in the industry in view of the long-range method of educating young men to enter this industry. Mr. Calkin said he was gratified at the support the Foundation has received from both pulp and paper mills and the allied industries.

On Saturday morning, May 17, Chairman Knudson presided at a brief business session of the New England Section of TAPPI at which the following officers were elected:

Chairman—H. W. Knudson, Hollingsworth & Vose Co.
Vice-Chairman—F. S. Klein, Byron Weston Co.
Treasurer—H. T. Barker, Bird & Son, Inc.
Secretary—Paul D. Applegate, Hercules Powder Co., Holyoke, Mass.
Executive Committee—Helen U. Kiely, American Writing Paper Corp.; Gordon Benson, St. Regis Paper Co.; S. J. Quattrocchi, Deerfield Glassine Co.; J. T. Loomer, Robert Gair Co.; George Soyka, Fort Orange Paper Co.; C. I. Horton, R. T. Vanderbilt Co.; George R. Wallace, 3rd, Fitchburg Paper Co.; Freeman Perry, Chemical Paper Mfg. Co.

"The Practical Man and the Technical Man" was the theme of a most interesting and informal discourse by Albert E. Bachmann, Vice-President of the Missisquoi Corp., Past National President of TAPPI.

Atop the huge wooden horse at Toy Town is Mrs. Mabel Muzzy while Mrs. Clark Zitsmann and Miss Sandy Ronzello lend encouragement

In his opening remarks Mr. Bachmann stated flatly that in his long experience in the industry the practical superintendent has always impressed him as a man of great intelligence, with a wide knowledge of mill operation, production, and maintenance and unafraid of the acceptance of responsibility.

J. R. Calkin, University of Maine

A. E. Bachmann, Missisquoi Corp.

Mr. Bachmann stated that the object of his talk was to further the cooperation between the practical and technical men on the operating functions of a pulp and paper mill and he explored some of the phases of the early and now lessening frictions among them. He stressed the impact of practical technical men and the engineer on our industry in the past 30 yr. and spoke of improvements in pulp bleaching and machine coating as outstanding achievements, along with the development of the brightness tester.

"The practical men made all the initial improvements," said Mr. Bachmann, "and these developments enabled the mills to bring in the technical men to further these improvements."

Mr. Bachmann then described from his prepared chart the essential items involved under the heading of "Operating Functions" and "Improvement Functions" necessary to better control of such functions. Involved in these functions, the speaker said, are production, technical control, laboratory, and research. Mr. Bachmann stated his belief that the interest of control is best served when the practical man is in charge, with the technical man functioning under him.

"The Staff Function," said Mr. Bachmann, "under which improvements are developed, is not generally in use in this industry, yet it is a most important function. It involves also hiring, job evaluation, incentives, planning and scheduling, industrial engineering, material handling, and control system development.

"The development of standard practices is generally neglected. Yet it is the best method by which one knows how to do a particular thing."

Continuing with the list of functions under the heading of Staff Functions, Mr. Bachmann spoke briefly on Cost Engineering, Cost Reduction, Establishment of Standards, Process Engineering, Sales and Customer Contacts, Complaint Analysis, Grade Developments, Quality Improvement, Quality Production Standards, Experience Analysis, Teaching, Meetings, Supplier Contacts, Laboratory and Research Contacts, and Supervision of Laboratory and Research.

Mr. Bachmann closed his remarks with the observation that the practical man and the technical man both are of vital importance to the industry because the functions of pulp and paper manufacturing mesh their duties and responsibilities.

Chairman Alfred Montagna of the Connecticut Valley Division of the Superintendents Association expressed his pleasure at the success of this wonderful meeting and thanked Miss Kiely and Mr. Applegate for the splendid program they had staged. Mr. Montagna then presented Charles H. Reese, Vice-President in Charge of Manufacturing, Nekoosa-Ed-

wards Paper Co., Port Edwards, Wis., and Past National President, Superintendents Association, who stated that it was a great pleasure to be in attendance and he appreciated the warm welcome tendered him. Mr. Reese congratulated Mr. Bachmann on the excellent talk the latter had just completed and expressed agreement with Mr. Bachmann's viewpoint on the importance of the practical man and technical man working as a team.

The Cocktail Party sponsored by the representatives of the allied industries in attendance was held in the Playhouse, a colorful structure, which, augmented by the fashionably dressed ladies, made the affair brilliantly successful. Following dinner at the Tavern members and guests then returned to the Playhouse for the presentation of prizes to the winners at golf, bridge, and numerous drawings. Dancing at the Playhouse was the closing event.

C. I. Horton, Henry Mellen, and Ray Kilty ably managed the Cocktail Party and Chairman Alfred Montagna had charge of general arrangements of the meeting.

A special program for the ladies included luncheon on Friday, a motor trip to the Cathedral of the Pines at Rindge, N. H., and card parties. On Saturday their program called for a Putting Contest, cards, and sightseeing.

JOHN J. O'BRIEN, *Recorder*
Paper Mill News

Lake States

The sixth and final meeting of the Lake States Section was held at the Butte des Morts Country Club in Appleton on Tuesday, May 13, 1952.

The golf course was available for the day for guests and good weather favored the sizable group who took advantage of the golf. In the late afternoon a cocktail hour was sponsored by the Affiliates of the Lake States area.

Chairman A. P. Adrain opened the dinner meeting with an acknowledgment of the fine cooperation of the Affiliates through Mr. Willoughby and Mr. Lauterbach, who assisted in the program for this meeting. A magician, Ted Monnett, provided further entertainment following dinner.

Results of the election for the coming year's officers were given as follows:

Chairman—Herbert W. Rowe, Nekoosa-Edwards Paper Co., Port Edwards, Wis.
Vice-Chairman—Robert J. Seidl, Forest Products Laboratory, Madison, Wis.
Secretary—L. A. Moss, Whiting-Plover Paper Co., Stevens Point, Wis.
Treasurer—S. R. (Jim) Parsons, Consolidated Water Power & Paper Co., Wisconsin Rapids, Wis.
Executive Committee—Reinhold A. Vogt, Combined Locks Paper Co., Combined Locks, Wis.; Gilbert K. Dickerman, Consolidated Water Power & Paper Co., Wisconsin Rapids, Wis.; Karl W. Fries, Rhinelander Paper Co., Rhinelander, Wis.; Donald MacLaurin, Institute of Paper Chemistry, Appleton, Wis.

K. P. Geohegan, President of National TAPPI, gave a talk on the national organization and its relation to local sections. His talk follows:

It was kind of you to invite me to be with you tonight; and I appreciate the opportunity. This is your final meeting of the year and one at which, I suspect, you would prefer to keep away from anything very serious.

Yet you were considerate enough to ask me to talk on the way in which National TAPPI functions, knowing that that is the subject on which I could talk most easily. But to me, the functioning of TAPPI is a very serious subject; so I will tell you what I have decided to do.

At a recent meeting of the Delaware Section, I tried to describe the Executive Committee; how the members are chosen, the duties assigned to them, and some of their problems. If any of you are interested, it probably will be published in *Tappi*.

So, rather than repeat that tonight, I thought that I would give you a very informal and personal account of some of the problems of the Executive Committee and how the members are trying to solve them.

To give you the picture as it came to me when I was first asked to serve on the Executive Committee I hadn't the least idea what the committee was supposed to do. For the first year, it appeared to be pretty much routine; voting on changes in the bylaws, designating funds for particular purposes, approving the time and place of the functional meetings, hearing reports from the divisional chairmen and from the Chairman of the Committee on Local Sections.

The meetings were long and tedious, and it seemed to take much more time than necessary to make the decisions. It wasn't until I was given some responsibility of my own, that I began to realize what it all meant.

Then, they asked me to be Chairman of the Committee on Local Sections. What was I supposed to do? Help the Local Sections. How? That was up to me. They were a long way off, and how could I know what they needed and wanted?

I have mentioned all this only because it described the problems that face every member of the Executive Committee. It gives you some idea as to why it is often difficult to reach a decision that will best serve the interests of the members.

Take the question as to whether we should publish our own magazine. That was a hard one. Did the members want it? What sort of a magazine would it be? What would it cost? Would we have to increase the membership dues? The discussion went on for meeting after meeting.

In retrospect, it appears as if the answer should have been obvious. Consider what it is doing in bringing to each member news of all the local sections. Note the quality of the papers that appear in it. You all know of the award of merit that Mr. Macdonald received because of the way in which it is prepared.

But we didn't know that he could undertake the editorship of it in addition to his other duties, and make such a success of it. Nor did we know that Dr. West would consent to act as Chairman of our Editorial Board.

Other problems come up every meeting, although I will admit not many are as important as the one I just cited. But whether large or small, the members of the Executive Committee discuss them from one angle and then from another until they are satisfied that the final decision serves best the interests of our TAPPI members.

Now let us return to my original problem of how to find out what could be done to help the local sections. Let us see what steps have been taken to bring the local sections and the national organization closer together.

When the Nominating Committee selects candidates for the Executive Committee, they take care that all parts of the country will be represented. That makes it possible to appoint a member of the Executive Committee as a representative to each local section.

That gives a chance for the representative to bring to the section any news that he considers important. It also gives the section officers and members a chance to pass on to the Executive Committee any suggestions, criticisms, or requests for help. It is a sound arrangement and works well; but we felt that it was not enough.

So recently, two further steps have been taken. First, during the February Convention, the Local Section Chairmen (or other representatives) meet with the Chairman of the Local Sections Committee. Usually, the national President and Vice-President and other members of the Local Sections Committee are present. Here the problems of the local sections are discussed. As each representative brings up the problems of his section, he obtains suggestions and advice from the other local sections and from the national officers. Often the discussion results in a needed change in our bylaws or regulations.

The second step was to establish the practice of inviting the chairman of each local section, or his representative, to attend a meeting of the Executive Committee on the day preceding the February Convention. This gives officers of the local sections direct contact with the national organization. They become acquainted with the members of the Executive Committee and their problems; and they can ask for aid when it is needed.

I do not remember who suggested these steps. It was not I, although I wish that I could take the credit. Through the arrangements I have mentioned and the publicity given the section meetings by *Tappi*, I feel that there is a remarkably close relationship between the local sections and the national organization. There will always be problems, of course, but the means of solving them has been provided.

I wish that the situation were as satisfactory in regard to our divisions and committees. Here is another problem, and one to which the Executive Committtee is giving much thought and discussion. Many of our committees are operating extremely well but many are doing little, and some are completely inactive. This problem is harder to solve than that of the local sections. We have only ten local sections; but we have 50 committees and 90 subcommittees.

In order to help the committees with their work, we have reorganized our Research Appropriations Committee. It now con-

sists of a chairman and ten members, and in addition, the President and Vice-President of the Association, who are members automatically. The other members have been chosen from our TAPPI membership because of their broad knowledge of research. Appropriations for research are not made without the approval of this committee.

In order that committees that need help will not have to wait until a meeting of the Executive Committee, this committee has the power to make appropriations totaling a fixed amount, provided the committee is unanimous in its approval. If even one member dissents, the question must be brought before the Executive Committee. During 1951, eleven projects were approved, and one was refused. At present, we have two projects that were not approved unanimously and are being held for the next meeting of the Executive Committee.

This is fine so far as it goes. It gives aid to committees that have a sound and constructive program; but it does not help the others develop their own programs. It does not furnish the imagination and originality that are required to recognize the need for a research program and the possibility of carrying it to a successful conclusion.

Chiefly during the Fall, but also during the rest of the year, functional meetings are being held that are being sponsored by some of our divisions and committees. These are committees that are functioning well. They may need some financial assistance to complete research projects, but they need no help in developing their programs.

Other strong committees are those whose activities are localized in some of our sections. Living near each other, their members are able to meet frequently and not rely on meetings during the February Convention when their time is limited and every member has other interests that vie for his attention. I wish to refer again to the success of these committees.

Now, as an example of the working of the Executive Committee, I have just described one of the problems that is facing it today. I have stated the situation; but before I sign off, I would like to suggest a possible solution for your consideration. Not that I am convinced that this is *the* solution, but because I would like to know whether it appears logical to you.

When considering this, please bear in mind that what we are seeking is a way to develop in our committees that spark that originates ideas for research and that enthusiasm that carries them to a conclusion.

Recently the Ohio Section had the honor and the pleasure of being addressed by D. C. Everest, President of the Marathon Corp. I imagine that most of you have heard him, and all of you who have know that we had reason to anticipate an inspiring talk. We were not disappointed. But it was his opening remark that impressed me particularly, probably because I connected it with the problem I have just outlined. He said, "If you don't get people to thinking and arguing about some phase of a subject, you are sunk."

For our purpose, I believe we can go a step further and add that you must convert your "thinking and arguing" into action or the ideas that result are valueless.

It looks to me as if that is probably what is wrong with those of our committees that are not very active. They do not have enough time to think and argue, nor the proper atmosphere in which to do it. Many of them meet only once a year during the February Convention when the time is short, when they have many other things on their minds, or when they are anxious to finish so as to attend another meeting.

I have already remarked that those committees that are localized in some of our sections, are functioning well. I believe that they are successful because their members can meet frequently with plenty of time for discussion. After all, there is nothing like a friendly argument to clear the air and develop ideas.

Then why not reorganize those of our committees that now need strengthening, and center each in a local section where its interests are related to the interests of the section members?

There is the danger that such a committee would be too local in its viewpoint and not have the breadth of view required of a national committee. Then why not centralize about half of the members in a section where they can meet and have their discussions; and keep the rest of the committee informed by correspondence? The written comments and suggestions of the rest of the committee would assure a national outlook.

It seems to me that this would have added advantages both to the committee and to the section in which it was centralized. One or two section meetings each year would be related to the committee's problems and activities. The discussion after the meeting would give the committee additional ideas and valuable suggestions.

After the committee's work had progressed and had resulted in a report or a paper on the chosen subject, it could be presented at the section meeting. Then it could be submitted to Mr. Macdonald and to our Editorial Board. If the work was well done, it should qualify for publication in the Technical Section of *Tappi*. Papers presented in this way would relieve some of the congestion at the February meetings and yet would be available to all our members.

I have attempted to illustrate to you the kind of problems that concern the Executive Committee by describing to you one that now faces it. It is one that concerns me deeply because I feel that its solution is important to the future of TAPPI.

Does the solution I have suggested appear logical to you? Or have you another to suggest? I would appreciate sincerely any comments or ideas you would care to express.

Kalamazoo Valley

The Kalamazoo Valley Section held its annual "Fun Day" at the Gull Lake Country Club on June 5. Approximately 150 members and guests attended.

Golf and numerous prizes were furnished through the courtesy of suppliers. A committee consisting of James H. Fritz, Chairman, Clarence Bickel, Claude Fletcher, and C. H. Turner handled all arrangements and conducted not only the golf tournament but arranged for shuffleboard, croquet, etc.

Some of the golf winners were Jack Hayes, low gross; Joe Horacek, runner-up; Don Huyser, low net; Herb Faram, Merle Wilkens and Horrace Brunner won blind bogey prizes.

A banquet in the Club House followed the golf matches. Following this the annual election of officers was held. Officers elected for 1952–53 are: A. T. Luey, Sutherland Paper Co., *Chairman;* R. H. Hurst, Kalamazoo Vegetable Parchment Co., *Vice-Chairman;* Arthur Dean, Michigan Paper Co., *Secretary*, and Richard Trelfa, Watervliet Paper Co., *Treasurer*.

R. H. Hurst, *Secretary*

Southeastern

The last meeting of the 1951–52 year of the Southeastern Pulp and Paper Society was held at Fort Sumter Hotel, Charleston, S. C., with Chairman G. C. Kimble presiding.

Following dinner, L. F. Thompson, Manager of the West Virginia Pulp & Paper Co., extended a welcome to Charleston and to his company's mill. Highlight of the business session was a motion that was carried to petition the Executive Committee of the Technical Association of the Pulp and Paper Industry for a charter as a Local Section of that Association. Another motion that carried related to the appointment of a Recording Secretary and a Corresponding Secretary by the Executive Committee. Also carried was a motion that would permit only members of the Technical Association of the Pulp and Paper Association to vote or hold office.

Election of officers followed, with voting from the floor. Those elected for the 1952–53 term were: G. C. Kimble, *Chairman;* M. B. Pineo, *Vice-Chairman;* and G. E. Scofield, *Treasurer*. Appointments to the Executive Committee included R. C. Sproull, Program Chairman; C. L. Clark, Local Arrangements; J. E. Overall, Corresponding Secretary; and W. T. Brown, Recording Secretary.

Under the chairmanship of M. B. Pineo, the program was presented, which included the following papers:

1. "The State's Views of Stream Improvement by the Pulp and Paper Industry," by David B. Lee, Sanitary Engineer, State Board of Health of Florida.
2. "National Council for Stream Improvement Activities," by W. A. Moggio, Southern Resident Engineer and Project Leader, National Council for Stream Improvement, Louisiana State University.
3. "Stream Pollutional Aspects of the Kraft Pulping Industry," by David B. Smith, Associate Professor of Civil Engineering, University of Florida, Gainesville, Fla.

After presentation of the above papers, C. A. Shoudy, Superintendent of the West Virginia Pulp & Paper Co., described briefly the operations of this plant, and invited the members and guests to visit the plant the following morning.

Attendance at the meeting included 34 members and 51 nonmembers.

G. E. Scofield, *Secretary*

Delaware Valley

The last dinner meeting of the current season of the Delaware Valley Section was held at the Engineers' Club, Philadelphia, Pa., on May 22, 1952. Section Chairman, August S. Erspamer, presided. There were approximately 80 members in attendance.

Mr. Erspamer extended his appreciation to the members for the excellent cooperation which they had extended to him during his tenure of office. He pointed out that the past season had been one of the best in every way. Mr. Erspamer then introduced the newly elected officers for the coming season. They were as follows:

Chairman—Harold C. Brill, E. I. du Pont de Nemours & Co.
First Vice-Chairman—Frank J. Lovegren, W. C. Hamilton Co.
Second Vice-Chairman, Emil R. Padavic, Container Corp. of America
Treasurer—Edward C. Molin, Glassine Paper Co.
Secretary—John J. Steinbinder, Riegel Paper Corp.

After the introduction the meeting was turned over to the new Chairman, Harold C. Brill, who announced that the program for the night was the presentation of four of the five papers submitted for the Albert Award competition. Fred Leser, Chairman of the Award Committee, then took over the meeting. He explained that final judging of the winner depended upon the presentation to be made. He pointed out that due to the time limitations only the best four of the five papers could be presented All participants received a copy of Suttermeister's "Chemistry of Pulp and Paper Making."

The contestants and their papers were as follows:

1. "White Water Utilization," by P. J. Schmitt, P. H. Glatfelter Co.
2. "Continuous pH Control on a Glassine Machine," R. C. Peterson, Riegel Paper Corp.
3. "Technical Aids in a Paper Mill Felt Program," by Miss N. Willenbecker, Scott Paper Co.
4. "Air Heater and Economizers in Paper Mill Power Plants," by W. C. Marble, Riegel Paper Corp.

Mr. Marble was selected unanimously as the winner with Mr. Schmitt a very close second. The plaque will be engraved with the winner's name and placed in his possession for one year. The winner also received a check for $50.

The meeting was adjourned after Mr. Albert expressed his appreciation for the honor of having the annual award named for him.

John J. Steinbinder, *Secretary*

The Application of Air Heaters and Economizers in Paper Mill Power Plants

William C. Marble

The installation of apparatus in boiler flues to recover part of the heat which passes out the stack in the gaseous products of combustion has been practiced for 90 years. Economizers were used in 1860 and air heaters in 1881(*1*). Although this equipment has long been considered essential in central station practice, its acceptance by industry is far from universal, and it is, therefore, the purpose of this paper to acquaint members of the paper industry with the characteristics, capabilities, and advantages of air heaters and economizers in paper mill power plants.

AIR HEATERS

The two classes of air heaters in common use are the recuperative and regenerative. In the recuperative class the most common is the tubular in which heat is transferred from the flue gases through the walls of tubes to the combustion air. They are usually constructed of 2 or $2^1/_2$-in. O.D. no. 14 gage steel tubing rolled into steel tube sheets with this assembly enclosed in an insulated steel casing which forms part of the boiler flue. Either the gas or the air may be passed through the tubes with gas inside the more frequent arrangement. The Ljungstrom regenerative type is made up of a series of corrugated steel sheets arranged in sections like pieces of pie about the longitudinal axis of a cylinder to permit a vigorous scrubbing of their surfaces by the flow of air or flue gas. These plates are enclosed by a cylindrical steel shell, and the assembly is rotated continuously by a small motor so that the plates alternately absorb heat from the flue gases and give it up to the air. Seals are provided to prevent excessive dilution of the flue gases by air infiltration. This infiltration is guaranteed to be not more than 10%, and approaches 6 to 7% in actual practice.

Other types of air heater are the plate type which differs from the tubular in that it is constructed of narrow, flat, steel envelopes through which heat is transferred to the air, the steam coil type which is similar to the blast heater used for space heating, and the direct-fired type in which the products of combustion of gas or oil are blended with low-temperature air and then supplied to the main furnace as part of the combustion air, and the separately-fired type which has its own furnace to provide hot gases for a recuperative or regenerative air heater.

The first cost of tubular air heaters is low. They require considerable space for installation, and in order to reduce this space requirement, the loss of draft and air pressure must be kept higher than in the other types. As with all the heat recovery class air heaters there is difficulty with corrosion in the low-temperature zones when burning high sulphur fuels. If retubing becomes necessary, the labor cost is high, but usually no special shapes are involved, and replacement costs are held down.

The Ljungstrom regenerative type is more expensive then the tubular air heaters, but economical of space, draft loss, and air pressure loss. Where high recoveries are desired the economy of space gives a definite advantage to this type. These units are constructed with a shallow layer of elements on the "cold end" in order that they may be easily removed for cleaning or replacement. In some public utility plants these "cold end" elements are removed from the boilers which have been shut down for the week end, and replaced with a spare set which has been purchased in factory assembled "bundles" or "baskets." The set which has been removed is washed at some convenient time to remove corrosive deposits and made ready for reinstallation at the next shutdown. Corrosion is important because of the high cost of spare parts, but otherwise, it is not as critical as in the plate and tubular types. For instance, this air heater may be operated after the elements have had holes eaten through them without dangerously reducing the amount of air supplied for combustion because of excessive leakage into the gas stream, at a stage when plate and tubular heaters would have to be taken out of service.

Efficiency of heat recovery would, of course, be reduced.

Plate air heaters will give performance equivalent to that of tubular air heaters with a lower draft loss and air pressure loss, and in a smaller space. Rising costs have, however, made their fabrication expensive, and their popularity has declined sharply.

ECONOMIZERS

The first economizers were installed in low-pressure boilers and in plants where there was no water treatment. Consequently, to resist the corrosion from oxygen dissolved in the feedwater, cast iron, which was suitable for the pressures of the day, was used for the tubes. Economizer arrangement was generally with vertical tubes and water flowing upward

William C. Marble, Engineer, Riegel Paper Corp., Milford, N. J.

making one pass through the gas stream. The use of higher and higher pressures and the desire for the more efficient arrangement of counterflow have led to the almost universal utilization of steel tube construction at the present.

When the economizer tubes are arranged in vertical banks with both ends in drums the unit is called an integral economizer. It is adjacent to the boiler tubes and enclosed in the

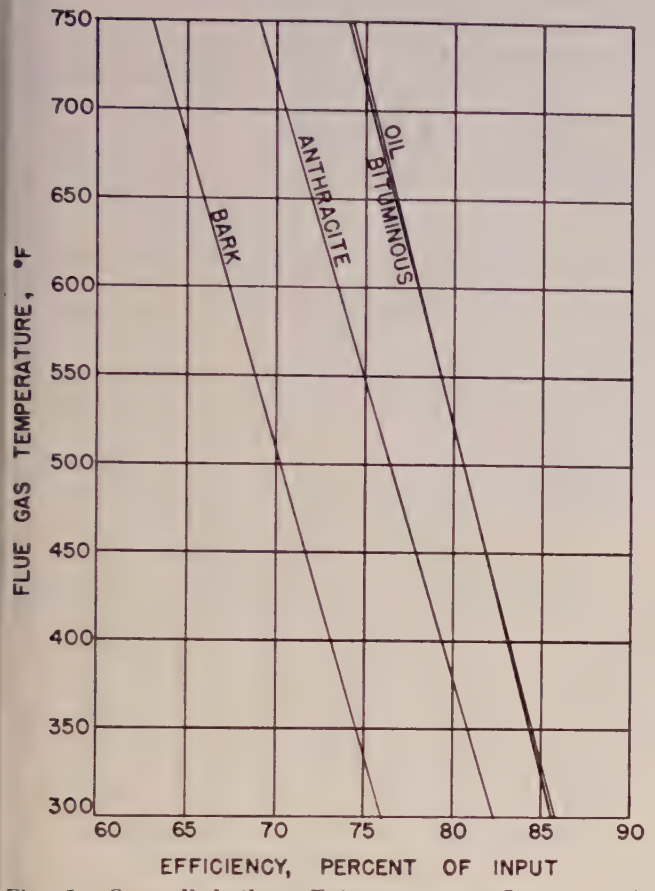


Fig. 1. Over-all boiler efficiency versus flue gas exit temperature

boiler setting. Such economizers, although they may be baffled for counterflow operation, have a relatively large water-flow area with resulting low velocities and poor distribution.

Today, most economizers are arranged with their tubes horizontal and gas flow vertical. Designers prefer upward flow of water as an aid in preventing steam pockets, and increase the water velocity and consequent pressure drop if conditions require downward flow of water. The steel tubes may be fitted with fins or rings to increase the heating surface per foot or left bare. One type which is particularly resistant to both corrosion and erosion employs cast-iron "gilled ring" sections shrunk on and completely encasing a steel tube. Economizer tubes were originally straight and fitted with a junction box or return header at each end for cleaning and inspection. As soon as it was realized that cleaning would not be affected, U tubes and bifurcated elements were utilized to reduce costs and the number of joints to be maintained. With modern feedwater treatment and the growing popularity of cleaning boilers with acid, many economizers of the continuous tube, all-welded type are now installed.

Considerations

Although much of the fly ash has been deposited by the flue gas by the time it reaches the heat recovery section of the steam generator, ash accumulates on the heating surfaces in this section, and suitable provision for cleaning them while the boiler is in service is essential. This is particularly true of the low-temperature section of air heaters, because with the high air velocities customarily encountered, the metal temperature tends to approximate that of the air, and condensation of the moisture in the gas takes place. Ash accumulations in this zone not only act as insulation and further lower these metal temperatures, but are also porous enough to permit infiltration of more moisture-carrying gas. If corrosive compounds are present in the gas or ash, the attack on the metal is accelerated, and there is a marked increase in maintenance. With the Ljungstrom air heater, the build-up of dirt on the heating surfaces causes an increase in fan power required, but, because of the regenerative principle, does not seriously affect the recovery of heat.

The effect of soot on heating surfaces through which heat must be transferred from one fluid to another as in economizers and the recuperative air heaters, however, is obviously detrimental. Air bypasses and recirculation are sometimes necessary to maintain adequate metal temperatures at low loads.

As regards the fuel fired, the ash and sulphur content have the greatest influence on the design of heat recovery equipment. Anthracite coal and bark are generally considered abrasive fuels and gas velocities are kept low with a resulting increase in space requirements. The high-sulphur Midwest bituminous coals and vanadium-bearing fuel oils require higher gas exit temperatures to prevent corrosion.

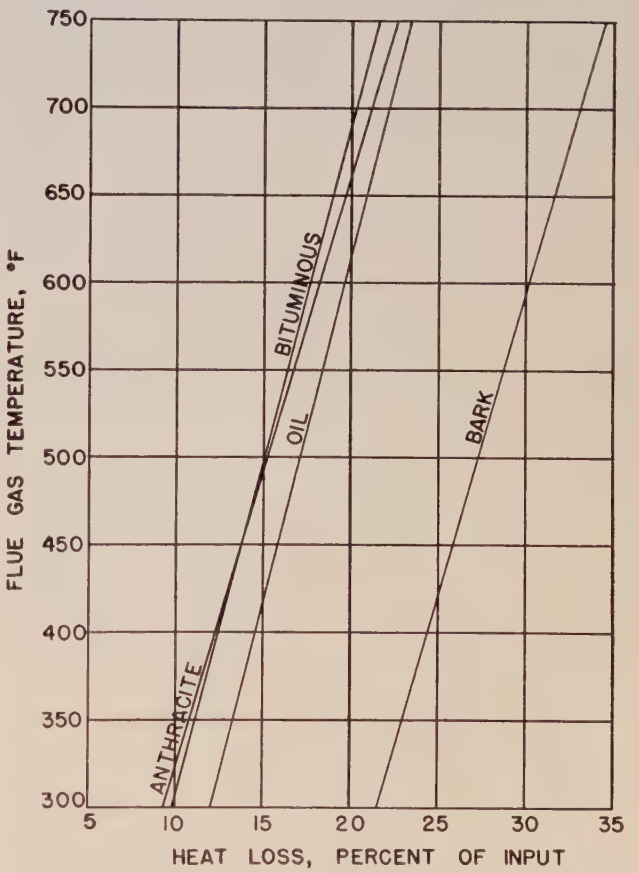


Fig. 2. Per cent of heat input lost in flue gases at various flue gas exit temperatures

The firing equipment also influences the selection of heat recovery apparatus. Most stoker manufacturers prefer a 300° maximum limit on air temperature, but will occasionally raise this to 350°. Primary air temperatures for pulverized fuel installations are regulated by the moisture in the coal fired, and are usually determined as part of the original design. With bark and fuel oil, air temperatures in the neighborhood

of 600° are common, and sometimes as high as 700°. With pulverized fuel, the primary air heater is located in a hot gas zone because of the high flue gas temperature required, and further reduction of the flue gas temperature is accomplished with a secondary air heater or an economizer. In some cases it may be possible to heat the total air to the temperature required for drying the coal and withdraw a portion for primary air. An air heater or economizer alone may provide sufficient recovery or the air heater may be placed downstream of the economizer, depending on the permissible or required air temperature. Each installation must be studied with regard to existing conditions, and a reputable manufacturer of this equipment will be able to recommend the best arrangement for the particular job.

The installation of heat recovery apparatus on new steam generating units is easily taken care of in their design, but its application to existing units requires the advice of a competent engineer, and usually the boiler manufacturer. The addition of an air heater, for example, may increase the furnace exit temperature to the point where an undesirable slagging condition occurs and additional furnace cooling may be required. The installation of an air heater or economizer will increase the necessary fan capacity, lower the final steam temperature at a given rating, and if the unit is operated at the increased capacity then available, the capacity of steam drums and internals may be exceeded. Space must be provided for the installation and removal of economizer elements and air heater tubes. An increase in feedwater pressure may be necessary to overcome the extra pressure drop through an economizer. Manufacturers do not recommend feedwater temperatures entering economizers of less then 200°F. nor water temperatures leaving which are closer than 35°F. to the saturation temperature at drum pressure. Of course,

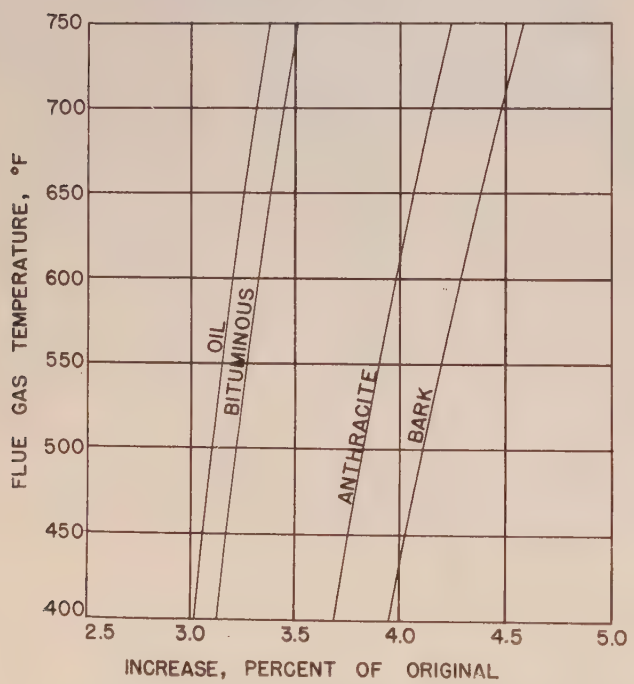


Fig. 3. Increase in steam generating capacity for 100°F. reduction in flue gas temperature from various gas exit temperatures

the cost of installing and operating the equipment must be evaluated against the anticipated benefits.

Evaluation

The examination of the capabilities of air heaters and economizers to increase the efficiency and capacity of a boiler has been limited to the following conditions:

Pressure at superheater outlet	400 p.s.i.
Final steam temperature	600°F.
Feedwater temperature	210–250°F.

Since it is beyond the scope of this paper to cover every variation of pressure and temperature, the above conditions were chosen as fairly representative of numerous "middle-

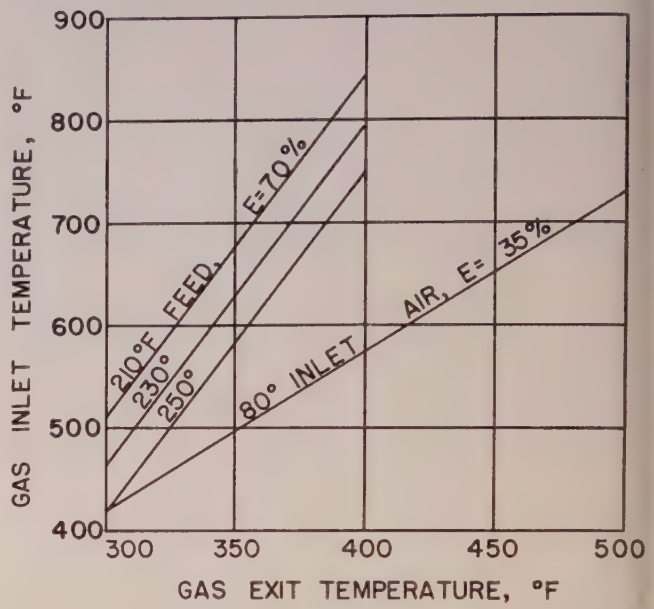


Fig. 4. Flue gas inlet temperatures for various flue gas exit temperatures for 70 and 35% gas temperature efficiencies

aged" boilers in plants which are feeling the pressures of increased fuel costs and greater steam demands. Calculations for the following data are based on the fuel analyses and combustion conditions listed below.

Proximate %		*Ultimate %*	
Pennsylvania Anthracite			
Vol.	4.81	C	69.06
FC	69.57	H_2	1.86
Ash	13.12	O_2	2.11
H_2O	12.50	N_2	0.78
S (sep.)	0.65	S	0.57
B.t.u./lb. as fired	10,970	Ash	13.12
		H_2O	12.50
Excess air at boiler outlet			40%
Loss due to unburned combustible			6%
West Virginia Bituminous Coal			
Vol.	34.31	C	70.12
FC	50.10	H_2	4.70
Ash	9.59	O_2	5.55
H_2O	6.00	N_2	1.41
S (sep.)	2.80	S	2.63
B.t.u./lb. as fired	12,878	Ash	9.59
		H_2O	6.00
Excess air at boiler outlet			30%
Loss due to unburned combustible			2%
Fuel Oil			
C	84.0	B.t.u./lb. as fired	18,500
H_2	12.7	Excess air at boiler outlet	20%
O_2	1.2		
N_2	1.7		
S	0.4		
Bark			
H_2O	55.00	B.t.u./lb. as fired	4119
C	22.60	Excess air at boiler outlet	30%
H_2	2.79		
N_2	0.02		
O_2	19.42		
Ash	0.17		

Efficiencies were calculated in accordance with the Manual of Industry Standards of the American Boiler and Affiliated Industries. "Unaccounted for" losses were taken as 1.5% and radiation loss as 0.9%, corresponding to a boiler of 100,000 lb. per hr. capacity with one water-cooled wall operating at full load. Unless otherwise noted, "flue gas temperature" refers to the temperature of the flue gases after they have passed all the installed heating surface.

Figure 1 shows the relationship between the over-all efficiency of the boiler and flue gas temperature, and Fig. 2 the percentage of heat input lost in the flue gases for various flue gas temperatures. In calculating over-all efficiency, the loss due to unburned combustible was assumed to be 6 and 2% for anthracite and bituminous coals, respectively. From the over-all efficiencies of Fig. 1, it can be shown that for each 100°F. decrease in flue gas temperature by recovering heat which is subsequently used for generating steam, the efficiency of the boiler is increased 2.93% when burning anthracite coal, 2.89% for bark, 2.60% for bituminous coal, and 2.51% for oil. The increase in the capacity of the 100°F. reduction of flue gas temperature is shown in Fig. 3.

Referring to Fig. 1, if the flue gas temperature of an oil-fired boiler operating with an output of 100 million B.t.u. per hr. were reduced from 600 to 350°F., the over-all efficiency of the unit would be increased from 78.0 to 84.5%, and the saving in fuel costing 35 cents per million B.t.u. at the burners would be over $25,000 for a 300-day operating year.

As with other equipment the benefits derived from the installation of heat recovery apparatus must be justified by comparing them with the cost of owning and operating it. Where some special requirement has to be met such as the heating of primary air for drying coal, the problem is simplified. Otherwise, the costs of the equipment must be more than balanced by the saving in fuel or the gain in steam capacity of the boiler. In the installation of complete new steam generators, the designer may proportion the water wall, superheater, boiler, air heater, and economizer surfaces to arrive at the most economical arrangement, but for the application of air heaters and economizers to existing units, he is confronted with a set of initial conditions over which he has no control.

A useful criterion for making an approximate selection is the gas temperature efficiency, the ratio of the flue gas temperature drop from inlet to exit of the equipment to the difference between the initial temperatures of the flue gas and the heated fluid, of the apparatus considered. Although each case must be treated separately, as a "rule of thumb," the maximum economical gas temperature efficiency of recuperative air heaters is about 35%, of economizers about 70%, and of regenerative air heaters about 65%. This is subject to wide variation since in many instances a higher efficiency is readily justified, and in small units, where the cost of heat recovery surface is high, it may be prudent to omit such equipment entirely. High available draft and pressure losses are an aid to the designer since they permit higher mass flows which improve distribution and reduce the space required

Figure 4 shows the gas inlet temperatures for various exit temperatures with feed temperatures between 210 and 250°F. and ambient air at 80°F. for 70 and 35% gas temperature efficiencies, respectively. In this feedwater temperature range the temperature of the feed leaving an economizer which has a gas temperature efficiency of 70% is reasonably constant with gas exit temperatures of 300 to 400°F. Table I shows to the nearest 5°F. the temperature of the feed leaving such an economizer for the fuels previously mentioned, and the final temperature of air entering a 35% and a 65% efficient air heater at 80°F., for various flue gas exit temperatures. An air heater having a gas temperature efficiency of 65% would have gas inlet temperatures of 651 and 794°F. for gas exit temperatures of 300 and 350°F., respectively.

Table I. Temperatures[a] of Feed water and Air Leaving Economizers and Air Heaters for Various Flue Gas Exit Temperatures

Feed Entering 210–250°F., Air Entering 80°F.

Unit	Gas exit temp., °F.	Fuel — Final temperature of fluid heated, °F.			
		Oil	Bituminous	Anthracite	Bark[b]
Economizer, 70% temperature efficiency	300	285	285	290	300
	350	325	325	335	350
	400	370	370	385	410
Air heater, 35% temperature efficiency	300	225	240	225	350
	350	260	265	260	420
	400	295	305	300	485
	450	330	340	335	555
	500	375	385	380	640
Air heater, 65% temperature efficiency	300	515	525	515	900
	350	635	650	640	1120

[a] Temperatures are given to the nearest 5°F.
[b] 80% of total air.

To illustrate the saving in fuel due to the installation of heat recovery equipment, consider a stoker-fired boiler operating at 400 p.s.i., 600°F. total steam temperature, 230°F. feedwater, 80°F. ambient air, and a load of 100,000 lb. per hr. If the unit were burning bituminous coal costing $9.50 per ton at the stoker, and the temperature of the flue gas were reduced from 650 to 350°F., the over-all efficiency of the unit would be increased from 76.7 to 84.5%, and the fuel saving for a 300-day operating year would be $36,400. This could be done by reducing the gas temperature to 523°F. with an economizer which would increase the feedwater temperature to 280°F., and then heating the total combustion air to 300°F. which would further reduce the flue gas to 350°F. If it were desired to install an economizer only, the temperature of the water entering the boiler drum would be 334°F. and the gas temperature efficiency of the economizer 71.4%. Using a regenerative air heater only, reducing the gas temperature to 350°F, would heat the total combustion air to about 460°F., and reduction to 300°F. would give an air temperature of about 520°F. These air temperatures are too high for stoker firing, but if the unit were fired with oil the efficiencies would be approximately the same as with bituminous coal, and if we assume the same cost per million B.t.u., this may be used to illustrate the economies affected. The saving in fuel for the 300-day year for the latter condition would be $41,700. If this unit, operating with 650°F. gas at the boiler outlet, were burning pulverized coal and already equipped with a primary air heater to give 600°F. air entering the mill, the gas temperature leaving the air heater would be 578°F. Further reduction of the gas temperature to 350°F. with an economizer would raise the feed temperature to 310°F.

It may be difficult to calculate the saving in fuel cost resulting from the application of heat recovery equipment to boilers utilizing bark or other wood wastes as a fuel. Since the bark burner rarely furnishes enough steam for total requirements, it may, however, be evaluated in terms of the saving in fuel burned to make up the steam necessary for process. Assume a bark-fired boiler operating at 50,000 lb. per hr., 400 p.s.i., 600°F. steam temperature, 230°F. feedwater, 750°F. gas temperature at the boiler outlet and 80% of the total air heated to 600°F. This would give us a gas temperature of 465°F. leaving the air heater and an over-all efficiency of 71.6%. Reducing the flue gas temperature to 350°F. would increase the capacity of the unit to 52,000 lb. per hr., and reduction to 300°F. to 53,000 lb. per hr. If we assume that steam not supplied by the bark burner is furnished by the pulverized fuel boiler described above operation with a flue gas exit temperature of 350°F., a fuel cost of $10 per ton at the burners, and an over-all efficiency of 84.5%, a reduction in load of 2000 lb. per hr. would result in a saving of $7250 for a 300-day operating year, and a reduction of 3000 lb. per hr., a saving of $10,880 per year.

GENERAL COMMENTS

The foregoing is a brief discussion of the characteristics of heat recovery equipment and its place in the paper mill power plant. While some of the conditions chosen for evaluation represent a very narrow portion of the range of operation of power boilers, the basic considerations illustrated are sufficiently general to give management, engineering, and operating personnel a practical understanding of this apparatus, and to make a preliminary survey of its suitability in a particular power plant. For an installation to be satisfactory, a thorough engineering and economic study by competent personnel is necessary, and the manufacturers of the boiler, firing equipment, and fans should be consulted as to possible adverse effects to their equipment.

LITERATURE CITED

1. de Lorenzi, Otto, editor, *Combustion Engineering* (1949).

RECENT BOOKS

Pressman's Ink Manual. By George M. Halpern Director, Manhattan School of Printing, Bullingers Guides, Inc., New York, 1952. Cloth, 5 × 8¼, 182 pages. $6.00.

This is a handy reference guide prepared for practical pressmen. Of special interest is the chapter on ink and paper relationship, which describes the effect of bodies of inks on various types of paper. A chart is shown on which recommendations are given. The causes and remedies for certain ink problems are related. Other chapters deal with special inks, compounds, driers, vehicles, theory and classification of color, process printing and ink estimation. Appended is an excellent glossary of terms. A fine know-how book.

Pulp and Paper—Chemistry and Technology, Vol. II. By James P. Casey, A. E. Staley Co., Interscience Publishers, Inc., New York and London, 1952. Cloth, 6 × 9, 628 pages. $15.

The second volume of Jim Casey's excellent presentation is welcome. The two volumes represent a prodigious effort on the part of Mr. Casey who is Chairman of the TAPPI Nonfibrous Materials Testing Committee. The second volume deals with the properties of paper, in which he goes into considerable detail in interpreting the many properties and means of their test.

The use of statistics in relation to paper testing is well covered. Excellent chapters follow on pigment coating, printing, laminating, and pasting, internal treatment of paper with resinous materials, coating with resins, and a thorough discussion of resins. Abundant footnotes supplement the information furnished.

Mr. Casey's up-to-date book meets a real need.

R. G. M.

Chemical Engineering Techniques. By B. E. Lauer and R. F. Heckman, Chemical Engineering Department, University of Colorado. Reinhold Publishing Corp., New York, 1952. Cloth, 6 × 9, 450 pages. $6.00.

Dr. Lauer, a member of TAPPI and recently Chairman of its Vocational Training Committee, has prepared a book that will be well received by all who are interested in plant operation and technology. Most students of chemical engineering would have been glad to have this volume available when they were studying the subject the hard way.

The restraint of the authors in avoiding mathematics and chemical equations is remarkable and should encourage reading on the part of operating executives who have become somewhat hazy in the details. The novel presentation consists of describing a number of well-defined, interrelated, and easily classified techniques. These follow: Assembly of Materials (transportation and storage); Preparation of Materials (size reduction); Energy (sources, distribution); Reaction (measuring materials and reaction vessels); Separation and Purification of Materials; Distribution (packaging and measurement of qualities).

The originality of presentation and the useful workaday information applicable to process industries, including pulp and paper manufacture justifies its addition to the operating and engineering libraries of companies and individuals interested in production.

R. G. M.

From Trees to Paper, The Story of Newsprint. By Henry B. Lent. The Macmillan Co., New York, 1952. Cloth, 6 × 8¾, 149 pages. $2.75.

This is a popular educational book prepared for readers between 10 and 14 years of age. Mr. Lent is a professional writer who gathered material for this volume by spending time with foresters, engineers, mill executives, research men, and others connected with the Ontario Paper Co. and the Quebec North Shore Paper Co. Chapter headings suggest its contents: woods manager, fire rangers, the supply depot, lumberjacks, the flume, the power plant, the paper mill, guiders, chippers, paper machines.

Unlike some books dealing with manufacturing operations this volume represents quite good reporting rather than the wild guessing and misinterpretation so often found in books written by laymen for youth. It should be a good gift for a youngster who might be inspired to consider the industry as an interesting place of employment at a later date.

Color in Business, Science and Industry. By Deane B. Judd, Photometry and Colorimetry Section, National Bureau of Standards. John Wiley & Sons, Inc., New York, 1952. Cloth, 6 × 9¼, 401 pages. $6.50. (Canada $8.13.)

The authorship by Dr. Judd of a book having the above title is a recommendation in itself. The reviews of the text by such qualified color technologists as Dorothy Nickerson, K. S. Gibson, I. A. Balinkin, Walter Granville (Container Corp. of America), E. A. Stearns, our Carl A. Foss, and other participants in the Inter-Society Color Council (TAPPI is a member) add to its prestige. Special attention is given to visual psychophysics. As stated by the author, the key to color problems of the future is to be found in this concept.

The text deals with such subjects as color matching, spectrophotometry, colorimeters, color standards, color language, gloss, opacity, Kubelka-Munk analysis, and identification and formulation of colorants. A valuable book.

Paper Year Book—1952. 10th Ed. Edited by Frank Dysarz. Davidson Publishing Co., 405 E. Superior St., Duluth 2, Minn. Cloth, 8½ × 11½, 644 pages. $10.

The tenth annual edition (1952) of the Paper Year Book is larger than the 1951 edition and is a complete

revision, giving details of about more than 1000 kinds of paper and related items closely associated with the paper industry. More than 700 photographs and 100 charts are shown. A thumb-indexing scheme assists the reader in locating the main subdivisions such as coarse paper, fine and printing papers, paper testing and manufacture, paper markets, and paper manufacturers' addresses.

Modern Chemical Processes. Vol. II. By the Editors of *Industrial and Engineering Chemistry*. Reinhold Publishing Co., New York, 1952. Cloth, 8 × 11, 290 pages. $5.00.

This book is the second in a series describing major chemical and process industry operations. Readers of *Ind. & Eng. Chem.* are familiar with these collaborative reports. Among the subjects covered are: Chemicals from Fats, Reagent Grade Chemicals, Cane Sugar Refining, Food Yeast from Sulphite Liquor, Modern Sulphur Mining, Magnesia-Base Sulphite Pulping, Kraft Pulp Production, Kraft Papermaking, etc.

The Conservation of Ground Water. By Harold E. Thomas, U. S. Geological Survey. McGraw-Hill Book Co., New York, 1951. Cloth, 6 × 9, 327 pages, 4 maps. $5.00.

This book reviews basic hydrologic principles and data regarding our total water resources. It describes the experience in 35 states with groundwater development. The book was sponsored by the Conservation Foundation which was established in 1948 as an independent non-political organization to investigate facts and to educate. A large proportion of the text deals with actual water problems in several parts of the country.

Performance of Lubricating Oils. By H. H. Zuidema, Shell Oil Co. Research Laboratory, Wood River, Ill. ACS Monograph No. 113. Reinhold Publishing Co., New York. Cloth, 6 × 9, 250 pages. $5.00.

This book summarizes and evaluates the wealth of data on the performance characteristics of lubricating oils under various conditions. Performance characteristics include oxidation, bearing corrosion, etc., that are common to all applications. It is an excellent companion volume to Bondi's "The Physical Chemistry of Lubricating Oils."

LETTERS TO THE EDITOR

Drying Rates

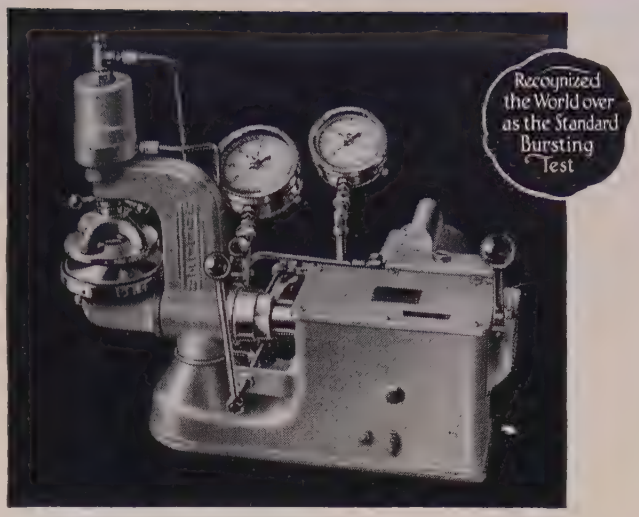
To the Editor, *Tappi:*

Do the TAPPI Data Sheets, Nos. 155 and 155G, apply for Yankee type machines, and can the curves be extended with any approximation to 80 p.s.i. steam pressure?

FRANK WINSHIP
Personal Products Corp.
Milltown, N. J.

To the Editor, *Tappi:*

The TAPPI Data Sheets Nos. 155 and 155G (and all the others, for that matter) do not apply to Yankee type driers where the intimacy of contact between the sheet and the drier surface gives a drying rate several times as high as is obtained on multicylinder driers.



MOTOR DRIVEN MODEL A TESTER. Especially designed for testing corrugated as well as other materials with bursting strengths above 200 pounds per square inch. Conforms to the test specifications of Rule 41 of the Consolidated Freight Classification No. 20. Special attachments available to quickly convert tester to perform flat crush tests or compression ring (edgewise) tests.

Send for booklet illustrating and describing the entire line of testers, and information on additional attachments.

B. F. PERKINS & SON, INC.
Holyoke, Massachusetts

The original drying rate report for tissue and absorbent papers, the reference for which is given on TAPPI Data Sheet 155, included a Fig. 3 giving the drying rate on Yankee driers, but at the time this was written in 1936, Yankee driers were not being operated at the high pressures common today, and the slope of the curves shown there should not be extrapolated to higher steam pressures. There is nothing in the literature to my knowledge giving the drying rate on Yankee driers. I may say, however, that in our experience on modern high temperature Yankee driers with 125 p.s.i. steam pressure and a good hood and air supply system several machines are obtaining a production drying rate R_p of 10 to 10.25 and an evaporation drying rate R_w of about 20. Accordingly, one might plot the drying rate on Yankee driers using the higher drying rate given in Fig. 3 of the report mentioned and extending the curve to the drying rate just given for 125 p.s.i. and arrive at a reasonable expectation of the drying rate of 80 p.s.i.

A. E. MONTGOMERY, *Vice-President*
J. O. Ross Engineering Co.
Chicago, Ill.

German Technical Association

To the Editor, *Tappi:*

In the May issue of *Tappi*, on p. 82 A, there appeared the names and addresses of technical associations, similar to TAPPI, located outside the United States.

There are some awkward distortions in the spelling of two words concerning the German association, as: (1) "Ingenieme" should be correctly: Ingenieure (meaning engineers); and (2) "Ludinigsplatz" should be correctly: Ludwigsplatz.

The German association would read in a freely translated

version: German Association of Pulp and Paper Chemists and Engineers." Its present secretary is Herr O. Schoeller.

Another distortion appears in the title of the French association. Instead of "Papetire" it should be: Papetière (with the accent grave on the "e" before the "r").

Emil Heuser
La Jolla, Calif.

Corrections—Tropical Woods of Western Africa

To the Editor, *Tappi:*

In the April, 1952, issue of *Tappi*, pages 174 to 178, in the article "Development of Chemical and Semichemical Processes for Deciduous Trees with Special Consideration of Tropical Woods of Western Africa" I wish to report some errors. In the headings of Table I and Table II mistakes have been made correction of which is necessary with regard to the importance of these tables. Therefore, I ask you to print the Tables I and II under "Letters to the Editor" once more in *Tappi*. The heading of Table I should be as indicated below. The sketches of cross sections in wood and paper in Table II should be as shown in the corrected table.

Referring to Fig. 1 on page 176 the densities of woods in grams per cubic centimeter must be: 0.2; 0.3; 0.4; 0.5; 0.6; 0.7; 0.8; 0.9; 1.0; 1.1; 1.2; 1.3;

In Table IV the breaking length of *Lophira procera* treated by the semichemical soda process must be 8550 instead of 85,500.

Roland O. H. Runkel
Bundesanstalt für Forst- und Holzwirtschaft
Reinbek/Hamburg, Germany

Bekk Smoothness Tester

To the Editor of *Tappi:*

In the May, 1952, issue of *Tappi*, page 154 A, it is stated that the Bekk smoothness tester is no longer available.

May we call to your attention that R. Fuess, Inc., 39 W. 60th St., New York 23, N. Y., is at present manufacturing this instrument in the United States. We have been manufacturing limited runs in the last five years in this country, as well as servicing the instrument.

Erwin Muscat
R. Fuess, Inc., New York, N. Y.

Starch on Single Facer

To the Editor, *Tappi:*

I wish to express my thanks for the copy of Mr. Vander Meulen's paper which was received today. This paper was one of the finest I have heard on the subject of starch use on the single facer. I look forward to your fine meetings which feature such interesting and informed speakers.

Warren E. Carlson
Lanzit Corrugated Box Co.
Chicago, Ill.

Japan TAPPI

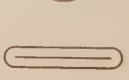
To the Editor, *Tappi:*

I have the pleasure of introducing myself as the new director-general of the Japan TAPPI, of which Tetsuo Okawa was re-elected as the chairman of board of directors at the fifth annual general meeting which took place in Tokyo on April 23, 1952.

Table I. Defibration of Hardwoods Treated with the Semichemical Soda Process

	Beech base concentration 3.15%			Maple base concentration 3.74%			Pine base concentration 3.74%			Spruce base concentration 2.55%		
Hr.	*Fibers, %*	*Shavings, %*	*Slime, %*	*Fibers, %*	*Shavings, %*	*Slime, %*	*Fibers, %*	*Shavings, %*	*Slime, %*	*Fibers, %*	*Shavings, %*	*Slime, %*
1	61.6	27.6	10.9	17.0	75.7	7.3	..					
1.25	71.5	18.2	10.0	26.0	62.7	11.3	..					
1.5	81.0	9.0	10.0	32.0	54.0	14.0	..	..				
1.75	85.7	4.3	10.0	45.4	37.4	17.2	..	..				
2	87.8	2.4	9.8	49.3	31.6	19.1	..	..	..	..	..	..
2.5	..	..	..	63.6	15.9	20.9	..	..	..	..	..	..
2.75	..	..	..	66.6	12.5	20.9	..	..	..	..	..	..
3	..	..	..	..	..	..	..	..	..	32.0	58.3	9.7
3.5	..	..	..	..	..	..	41.5	47.8	10.7	..	..	..
4	..	..	..	..	..	..	47.5	44.1	10.4	51.2	36.3	12.5
5	..	..	..	..	..	..	65.0	21.1	13.9	..	..	..
5.5	..	..	..	..	..	..	74.2	11.4	14.4	73.7	10.7	15.6
6	..	..	..	..	..	..	74.1	6.9	16.0	78.7	4.0	17.3
6.5	..	..	..	..	..	..	84.3	2.7	13.0	..	..	
Yield, %		82.2			85.2			82.3			82.7	

Table II. Morphological Nature of Fiber and Pulp Types

Wood Species	*Extremely Hard*		*Hard*		*Middle*		*Soft*			*Very Soft*	
	Diospyros	*Guajacum*	*Lophira procera*	*Rhizophora mangle*	*Sarcocephalus*	*Ptero carpus*	*Terminalia*	*Canarium*	*Pycnantus combo*	*Ceiba pentandra*	*Musanga smithii*
Weight per unit of volume	1.32	1.27	1.12	1.04	0.80	0.73	0.59	0.50	0.49	0.29	0.21
Cross section in wood											
Cross section in paper											
Cementation	Very slight		Slight		Middle		Good			Very good	
Strength (unbeaten)	Very small		Small		Middle		Good			Very good	
Breaking length, m.	...	...	1579	1565	...	1806	2520	..	5758	5019	5122
Folding strength	...	...	...	...	...	2	2	..	199	132	465
Opacity	Opaque		Opaque		Somewhat transparent		Transparent			Very transparent	
Beating			Fibrillating		Partly fibrillating, partly crushing		Partly fibrillating, partly crushing				

You will be interested to note some of the activities of your sister organization in Japan.

The Japan TAPPI at present claims an active membership of 2150 engineers and chemists, all engaged in the pulp and paper industry with 3 honorary members, 60 corporate members, and 49 supporting members. The number of active members shows an increase of 419 since a year ago.

Four lecture-meetings were sponsored by the TAPPI last year, April, June, and December meetings at Tokyo and November gathering at Sapporo, Hokkaido, each attended by a large number of individuals.

Various special committee meetings were held from time to time in discussing:

1. Pulp and paper testing.
2. Compilation of standard technical terms.
3. Over-all materials consumption unit for pulp manufacture.
4. Hardwood utilization.

The Japan TAPPI has also cooperated with the Soda Industry Association, the Japan Chemical Industry Society, the Paper and Pulp Federation, and Government Industrial Technical Bureau for the discussion of:

1. Quality standard of chloride.
2. Protection of marine and aquatic products resources.
3. Water supply problems.
4. Stream pollution problems.
5. Special treatment of taxation.
6. Laws expediting the rationalization of industry.
7. Tariff exempt of imported machinery.
8. Paper and pulp testing methods.

The following members were honored with prizes and awards for their outstanding contributions to the industry for 1952:

1. Yoshikuni Seki, Executive Director of Mitsubishi Paper Co. and ex-Director-General of the TAPPI.
2. Hidematsu Kaneniwa, Executive Director of Tomakomai Paper Co.
3. Takeshi Sato, technical staff of the TAPPI.
4. Shigeo Mayeno (Mitsubishi Paper Co.) for his paper on "Starch as Coating Material for Art Paper."
5. Hiroshi Asaoka (Tohoku Pulp Co.) for his paper on "Polymerization Degree of Pulp."

The presentation took place at a special meeting held in March, 1952.

The Japan TAPPI also edited during 1951, 7 issues of the *Pulp Industry Magazine*, its publicity organ, reporting various phases of the industry, one of which was a special issue dealing with articles contributed by the members who have made inspection trips abroad recently.

As 1952 activities, the Association plans to hold four lecture-meetings and one field trip; to publish a Japan TAPPI annual report; to compile a Standard "Technical Terms Text"; and to edit a special number commemorating the fifth anniversary of its founding in September, 1952, beside continuing ordinary research works.

The officials of the Japan TAPPI are a chairman, a director-general, two assistant directors-general (Haruzo Idogawa, Vice-President of Tohoku Pulp Co., and Hidematsu Kaneniwa, Executive Director of Tomakomai Paper Co.), and 18 more directors, one of whom is a paid managing director, and 2 auditors.

Such is a résumé of our organization.

SATOSHI YAMANOUCHI, *Director-General*
The Japan Technical Association
of Pulp and Paper Industry

PLANT ENGINEER; Southeast. To coordinate activities of design engineering firm and contractors during construction. Direct all engineering and maintenance in completed mill. Should have not less than ten years' experience in pulp and paper mill maintenance. Experience in maintenance of sulphate pulp production facilities necessary. Familiarity with groundwood and newsprint production desirable Summarize experience. Reply to *Tappi* P 216-52 122 E. 42nd Street, New York 17, N. Y.

EMPLOYMENT SERVICE

POSITIONS OPEN

P211-52. Quality Control Supervisor. Opportunity for security and advancement with well-established and growing organization for competent person experienced in paper converting and printing. Laboratory work and ink formulation experience helpful though chief demand is for application on the production line. Prefer graduate chemist. Write giving information concerning work experience, education, and relevant personal data. Replies confidential.

P212-52. Assistant Superintendent, Paper Mill. Technical graduate with minimum 10 years' paper mill experience. Minimum 5 years' experience in production supervision. Flooring and roofing felt mill, and building paper specialties. Two single-cylinder machines 160 tons per day. San Francisco Bay Area. Reply with résumé to Pabco Products, Inc., Emeryville 8, Calif.

P2135-2. An opening on the professional staff for a man capable of initiating and directing research in the chemical engineering aspects of the pulp and paper industry. The applicant would be expected also to take his share of the undergraduate instruction in the department. Write to R. R. McLaughlin, Head, Department of Chemical Engineering, University of Toronto, Totonto, Ont., Canada.

P214-52. Research Position. Paper chemist to organize and conduct research and development work on specialty papers for a paper mill located in the Middle West.

P215-52. Paper Chemist, to carry on technical research program in connection with specifications for papers used as card stock in IBM machines. Excellent working and living conditions, good salary, moving expenses paid, exceptional employee benefits. Challenging, permanent future.

POSITIONS WANTED

E176-52. Mechanical Engineer. 28. Desires position as Assistant to Mill Manager or Superintendent, Plant Engineer, or Maintenance Supervisor. Three and one-half years' paper mill experience. Present position 3 years as Plant Engineer in two-machine mill. Familiar with fourdrinier and cylinder machines and converting operations. Will consider any location.

E177-52. Pulp Mill Superintendent and former Chief Chemist with 12 years' experience in kraft and board mills desires comparable position with a progressive company. Familiar with multistage bleaching and some semichemical pulping. 36. B.S. degree.

E178-52. Chemical Engineer. 16 years' experience in board mills, corrugated and fiber containers, folding cartons. Plant Manager for last 6 years. Prefer Miami Valley Area.

E179-52. Chemist. Married. Experienced in general mill problems, process and technical control, felt and wire problems. Presently employed.

E180-52. Superintendent. 25 years' practical and technical experience on fine specialty grades. Experienced on both Yankee and fourdrinier machines, deinking and groundwood operations. Available on reasonably short notice.

E181-52. Graduate Chemist. 17 years' practical operating research experience in bleached sulphite and sulphate pulp and papers. Excellent employee relations. Desires position in operation or management.

E182-52. Sales Executive—paper school graduate. Over 12 years' experience in pulp and paper manufacture as technical director and assistant superintendent. Knows all phases of paper industry. Now in sales and development of paper chemicals. Desires similar job with greater future and remuneration.

WANTED: RESEARCH CHEMIST

Immediate opening in research and development department of specialty paper manufacturer. Excellent opportunity for young man with initiative and imagination. Experience or background in paper technology desirable but not essential. In first letter give complete resume of experience, age, education, and present salary. Address Technical Director, Hollingsworth & Vose Co., East Walpole, Mass.

Coating Ingredients from Tall Oil

J. W. ELDRIDGE and M. G. SELDEN

THE history of the use of tall oil as a coating ingredient is a long one. The literature and many patents state that products containing tall oil will dry in the presence of the usual drier metals, but the rate and type of drying are considered to be entirely impractical from the viewpoint of modern manufacture.

What are needed are full-fledged tall oil products that compare favorably with the vehicles made from the established drying oils including linseed and tung oils, and which can be produced in a few practical steps. And such a product is right this moment even closer than just around the corner! Now fortuitously, conditions are just right today for us, as an industry, to capitalize on such developments and establish a very profitable by-product business. For in these times when availability of raw materials has such a widespread influence, a program with a little imagination, backed by creative and organizing abilities, can place tall oil in broad markets permanently! Now, of course, a new substitute must be able to prove its worth, but present knowledge indicates no cause for worry here. Obviously, for our own good, both as individual companies and as an industry, we should always put on the market just as high quality a product as up-to-date technology and economics permit, and guard against marketing products which could send the reputation and price of tall oil products down instead of up.

PREPARATION OF MODERN VEHICLES

Before considering the manufacture of coating ingredients from tall oil, it might be well to look over the general field of established drying oils.

The value of a drying oil consists in its ability to form a satisfactory film over surfaces to which it is applied, and its ability to provide adequate viscosity, drying, and resistance properties when used as a varnish ingredient.

Linseed oil has been the most important paint vehicle ingredient since the early ages and still is of great importance at the present time. However, the raw linseed oil which was formerly used almost exclusively as a vehicle, and in the form in which it is produced by nature, is now being replaced by increasing quantities of specially treated and refined oils, such as acid-refined, alkali-refined, blown oils, and heat-polymerized oils. Thus, linseed is now considered to be a raw material which is improved by treatments and purification.

Tung oil is another established paint vehicle ingredient of great importance. Manufacturers were reluctant to use this material because it dried to a more brittle film and possessed frosting and wrinkling characteristics. Also, the oil was difficult to control as it readily polymerized to a gel. Later, it was found that by processing tung oil into varnishes at temperatures above 500°F., or by the use of certain phenolic-type resins in conjunction with the tung oil that these wrinkling and frosting tendencies were overcome, and very desirable properties could be obtained from its use.

The synthetic drying oils are based on creation of conjugate double bonds by various methods, and dehydration of hydroxyl acids. Examples of these are seen in the dehydration of castor oil and the polymerization of unsaturated hydrocarbons such as acetylene. The field of synthetic drying oils also includes simple esters of fatty acids and polyhydric alcohol which in some cases have been improved by conjugation, fractionation of the more unsaturated constituents, or by reacting with maleic anhydride.

Without going too deeply into the subject, it may be said that linseed and tung oils, as well as the other drying oils are different chemical individuals, and the properties of each depend upon the type and proportion of the constituent fatty acids making up the oil.

INFLUENCE OF RESINS

Also, and of greatest importance, it is standard practice to modify the properties of the established oils by cooking them with resins, which is somewhat similar to the conversion of cast iron into various types of steel by means of adding carbon, manganese, nickel. Furthermore, the properties of the resin-modified oils or the modified steel may be varied by changes in heat treatment.

The processing of a resin with an oil changes the characteristics of the oil. Under heat treatment the addition of one resin may increase the rate of bodying while a resin of another type may decrease it. The properties of the film formed by air drying or baking are likewise affected in such qualities as hardness, brittleness or flexibility retention on aging, waterproofness, alkali resistance, gloss, and in other ways. The introduction of a synthetic or natural resin into an oil, accompanied by heat processing, creates a different and exceedingly interesting type of vehicle which is called a varnish.

Tremendous progress has already been made in the development of oleoresinous as well as alkyd-type varnishes which dry rapidly and have desirable physical properties and durability. Thus, paint and enamel vehicles can be designed to emphasize any desired property by employing the right type and proportion of resin, accompanied by suitable processing. This, then, offers the opportunity to develop tall oil into products which can compete successfully and take their place among the established drying oils, varnishes, and paint vehicles.

TALL OIL VEHICLE DESIGN

Now, as is known, tall oil is not an ester but consists essentially of fatty acids and resin acids, together with some impurities. The fatty acids are believed to consist of about 48% linoleic acid, 45% oleic acid, and 7% saturated acids. If we combine only these fatty acids with polyhydric alcohols, we obtain esters which do not dry satisfactorily because the ester of oleic acid itself does not dry satisfactorily. However, the resin acids in tall oil are esterified at the same time as the fatty acids and are thus converted into a type of synthetic resin. The properties of both the resin formed by esterification of the resin acids and the oil formed by esterification of the fatty acids depend to a large extent upon the polyhydric alcohol used. Esters of tall oil made with pentaerythritol are much preferred to those made with glycerine because penta has four primary hydroxyl groups which are all reactive. Therefore, the penta ester has greater unsaturate functionality and dries more rapidly and to a harder film. An unmodified rosin ester of pentaerythritol has better drying properties than the unmodified rosin ester of glycerol, as well as greater resistance to water and alkali. In a similar manmer, pentaerythritol converts the resin acids contained in tall oil into products which dry better and possess improved water and alkali resistance. A still better drying and harder film can be obtained by introducing a small quantity of maleic anhydride into the tall oil. Abietic acid and its isomers make up the greater portion of the resin acids, but it does not react readily with maleic anhydride. By raising the temperature to about 400°F. the isomerization equilibrium is shifted toward levo-pimaric acid which readily forms the maleic adduct with the resin acids by the Diels Alder synthesis. When this new compound is esterified with penta, we have an ideal resin for

J. W. ELDRIDGE, Assistant Professor of Chemical Engineering, University of Virginia, Charlottesville, Va., and M. G. SELDEN, Chemist, Albemarle Paper Mfg. Co., Richmond, Va.

our purpose, and we have transformed a liability consisting of resin acids into a valuable asset.

OIL LENGTH

We then come to the quantity of resin contained in the tall oil. In other words, the proportion of resin acids to fatty acids in the tall oil begins to take on considerable importance as the length of the oil is controlled by this proportion. By oil length we mean the number of gallons of linseed oil, for instance, that is combined with 100 pounds of resin. Thus, it is possible to have 20 gallons of linseed oil combined with 100 pounds of resin, which is called a short oil length; or we may have 80 gallons of linseed oil combined with 100 pounds of resin, which is called a long oil length. Now, if we are working with a tall oil which contains, for example, 50% resin acids, we would require 200 pounds of tall oil to obtain 100 pounds of resin acids. We would then have 100 pounds remaining which would consist of fatty acids, if free from unsaponifiable matter. After esterification with penta, the weights of the esterified resin acids and fatty acids would be changed but the proportion remains approximately the same. Therefore, we may conclude that the above 100 pounds would produce the equivalent of 12.5 gallons of drying oil for each 100 pounds of our pentaerythritol synthetic resin. In a paint or enamel formula calling for a vehicle of this short oil length, the tall oil penta ester could be used to replace both the resin and the drying oil of the conventional formulation. If the paint or enamel formula called for a longer oil length, the tall oil ester could be used to replace all of the resin and part of the drying oil to the extent of its drying oil composition.

DEFECTS

Tall oil esters, having a color of 11 to 12 on the Gardner 1933 scale have been made and sold commercially for some time. However, as long as the esters possess the defects of the initial raw tall oil, under ordinary market conditions, the use is definitely limited. Even if we have a product that is outstanding in color and has good film properties, you may be sure that nobody will pay much for it, if it still smells of the pulp mill.

The defects of raw tall oil consist of sulphur compounds that produce offensive odors and react unfavorably with drier metals; color bodies that confine the use to darker products; and antioxidants which slow up the drying.

Various refining methods may be employed to improve tall oil such as acid refining, selective solvent refining, and distillation. Any of these improve the visible color. However, tall oil is noted for its poor color stability. In other words, refined tall oil darkens upon standing. This indicates the presence of potential color bodies which cause later discoloration.

Now, we all agree that these defects really are serious and that they present a rather formidable problem when an attempt is made to use so-called refined tall oil which has been only partially refined. If we are to be successful in making a strictly first class coating ingredient which is essentially equivalent to vehicles made with the established drying oils, the defects must be eliminated, and they can be by proper refining techniques, which are both practical and economical.

ACCEPTABLE VEHICLES

At the present time, products having the required standard of quality are being developed. Samples of an ester, which we have been able to work out, equal bleached linseed in color and have good color stability. The oil length is 20 gallons long. With the conventional quantities of drier metals it dries, sets to touch in 1 hour, and tack free in 16 hours. The air-dry film has a Sward hardness of 108, and exhibits good resistance to alkali and water.

As we succeed in removing the objectional defects, tall oil can be used by hundreds of small manufacturers who do not have the facilities and technical help to refine it themselves. In this way, and in this way alone, we can hope to build a reputation for tall oil and create a vigorous demand at all times.

For years, crude tall oil has been consistently sold for about 2 cents per pound, making it one of the cheapest organic acids available. The price has been based upon the cost to produce it and is in no way connected with the value of the material which it replaces. Our low cost of production is a powerful basic advantage and it is our job to see that not too much of this advantage is lost in refining, for a high refining cost would be a serious handicap to the initial expansion of the program, as well as a robber of profit. It is our firm conviction, however, that refining costs can be kept well within reason.

Now a word as to the potential profits, which are, of course, the motive for this program. It will undoubtedly prove that *at first*, tall oil products even when highly refined, will have to be generally priced as substitutes; that is, appreciably lower than the materials they replace. But once the reputation of these products has been firmly established, then there is no reason why they can't be sold at prices competitive with their competitors, linseed, tung oil, etc., and *therein lies a real profit!*

In conclusion then, we now know that really *good* coating ingredients can be made from tall oil, and we believe the time is right for our industry to start converting this by-product, tall oil, into some very substantial profits!

RECEIVED Aug. 30, 1951. Presented at the Fifth Alkaline Pulping Conference of the Technical Association of the Pulp and Paper Industry, Richmond, Va., Sept. 26–28, 1951.

Sorry! We should have said—
"32,400 cuts per MINUTE"
in our last advertisement.

Our "Multiplex" rag cutters vary in number of knives on the revolving knife head and the number of bed knives in the cradle and the RPM of the knife cylinder.

With six cylinder knives, one fixed bed knife, and eight auxiliary cradle knives 32,400 cuts per *minute* would be given rags with the normal head speed of 600 RPM.

Please ask us for a copy of our latest folder describing Taylor-Stiles cutters used by the paper industry.

TAYLOR, STILES & COMPANY
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TAPPI DATA SHEETS

Engineering, Chemical, and Operating Data

Digester Inspection Report Data Sheet

SUBMITTED BY THE DIGESTER CORROSION COMMITTEE

THIS relates to a procedure for the acquiring and recording of data pertaining to the corrosion of alkaline digesters and to the service life of the vessel.

The objectives of the data sheet are:

1. To establish an accurate and representative rate of corrosion of the digester material of construction.
2. To record this information on a form which lends itself to the intelligent analysis of the data.
3. To utilize the data obtained in checking the safety of the vessel, in replacing the vessel, in establishing digester costs, and in selecting a more economical material of construction.

A. DETERMINATION OF WALL THICKNESS

Method of Measurement

It is recommended that a suitable instrument be used of which there are currently available several varieties capable of measuring nondestructively the wall thickness of the vessel with considerable precision. Some of these instruments are applicable to carbon steel and some particularly applicable to nonmagnetic metallic linings or cladding. The various manufacturers of these instruments are prepared to give specific instructions as to the operation of their respective instruments.

For further discussion of this subject see paragraphs UA-804 through UA-807 of the ASME Boiler Code for Unfired Pressure Vessels for 1950.

Personnel

It is recommended that an individual or group of individuals be assigned to operate the measuring instruments. In this way, personal error factors will tend to be minimized.

B. SAMPLING AND PROCEDURE

Generally, a reproducible grid is laid out on the interior surface of the digester using some fixed reference point, such as a nozzle or a weld, so that the points of measurement will be exactly the same each time the digester is inspected. The point of measurement is arbitrarily the lower left-hand corner of the grid rectangle. The measurements are recorded on a suitable form, see Fig. 1 for illustration. This sheet should also show, for reference, location of seam and repair welds, all fittings and strainers in the digester.

Notations are made as to wear pattern, scale formation, localized corrosion, repair areas, points at which photographs or wax impressions were taken, and other items of interest having direct relation to digester corrosion and/or service life. These notations are recorded on a suitable form, see Fig. 2 for illustration.

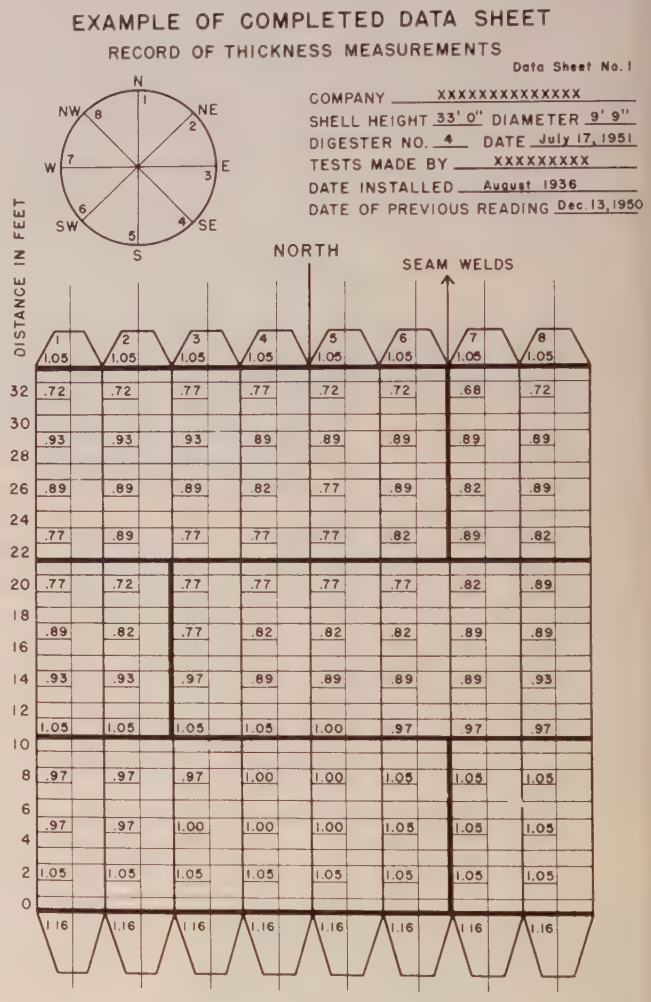

Fig. 1

It is to be noted Figs. 1 and 2 can and should be modified to fit the physical dimensions of the digesters being inspected.

It is recommended that a wall thickness survey and general inspection be made prior to placing the vessel in service and at intervals of approximately 6 mo. thereafter.

C. SUMMARY REPORT FORMS

A recommended form, Table I, may be used for sum-

marizing the information and data obtained in "A" and "B" above. The use of this summary form will permit a quick and accurate appraisal of the current status of each digester.

Table I. Summary of Digester Inspection

A. Basic Reference Data
- (1) Digester number
- (2) Report date
- (3) Date this inspection
- (4) Date last previous inspection
- (5) Date put in service
- (6) Vessel manufactured by
- (7) Manufacturer's serial number
- (8) Purchase order number
- (9) Steel spec. ASTM number
- (10) Nominal original thickness
  - (a) Top
  - (b) Shell
  - (c) Bottom
- (11) Present operating pressure, p.s.i.g.
- (12) Number cooks to date
- (13) Number cooks since last inspection

B. Critical Corrosion Data
- (1) Location of area on which estimated remaining life is based
- (2) Thicknesses this area, in
  - (a) Present inspection
  - (b) Previous inspection
  - (c) Minimum allowable based on ____ p.s.i.g. operating pressure
- (3) Current corrosion rate this area, *IPY*
- (4) Estimated remaining life, yr.

C. Remarks

By comparing the results reported on each of the three report sheets, one can obtain a picture of how each digester is corroding. The remaining service life of the digester can be estimated and, hence, digester replacements can be planned. Further, one will be able to evaluate digester costs on a per ton of pulp production basis and possibly decide to change the material of construction to obtain a lower cost per ton service life. In addition to these advantages, the very important possession of accurate knowledge will permit a more realistic approach to the safety aspects of operating a pressure vessel.

D. ESTIMATED REMAINING LIFE

Estimated remaining life of a digester should be the best unbiased estimate of the time until the vessel must be removed from service because of corrosion. The following expression should be used:

$$L = \frac{t_1 - t_2}{IPY}$$

where

L = estimated remaining life, yr. (Table I, B-4)
t_1 = present thickness, in. (Table I, B-2a)
t_2 = minimum allowable thickness, in. (Table I, B-2c)
IPY = corrosion rate, in./yr. (Table I, B-3)

Each of these quantities should refer as specifically as possible to the critical area where failure is most likely to occur:

(t_1) Serious failures are liable to occur, not at a point or an isolated pit, but in an area of some magnitude. Where significant loss of metal is indicated, by visual inspection or by thickness records, such as Fig. 1, additional nearby measurements should be taken to determine the extent of the corroded area. The following rules are suggested: (1) In an area over 1 sq. ft., use the least thickness. (2) In an area under 1 sq. ft., use the average thickness (see API-ASME Code, 1943, I-205, also ASME Code 1950, Par. UA-806b and c).

(t_2) Refer to insurance and applicable code requirements, based on specific operating pressure.

EXAMPLE OF COMPLETED DATA SHEET

RECORD OF VISUAL INSPECTION Data Sheet No. 2

COMPANY XXXXXXXXXXXXXX INSPECTED BY XXXXXXX
DATE July 17, 1951 DIGESTER NUMBER 4

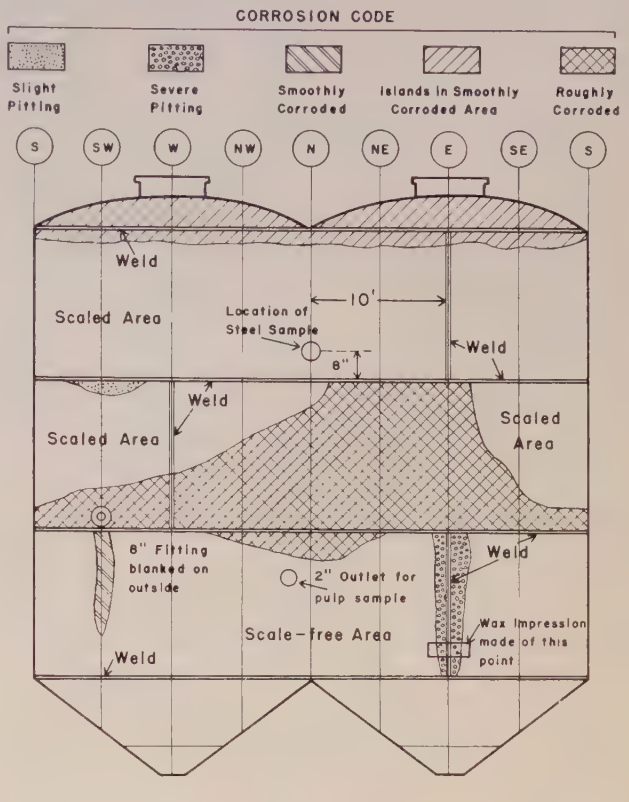

Fig. 2

(*IPY*) Ordinary random errors in thickness measurement, if only two such readings are compared, can produce large deviations in apparent corrosion rate. With every additional consecutive measurement available for the *same* point, progressively improved precision is obtained. It is important that all available thickness records pertinent to the critical area should be carefully analyzed to obtain the best unbiased estimate of the current corrosion trend. It is this value of *IPY* which should be used in the above formula.

SEVENTH ENGINEERING CONFERENCE

of the

Technical Association of the Pulp and Paper Industry

Morrison Hotel, Chicago, Ill.
Oct. 13–16, 1952

Instrumentation Studies. LXXII. Methods of Measuring the Moisture Content of Paper

II. The Fielden Drimeter

J. A. VAN DEN AKKER and K. W. HARDACKER

The Fielden Drimeter is an electrical instrument designed to indicate the moisture content of a moving web of fabric or paper through the partial dependence of the dielectric constant of the combination of fiber and water on the moisture content. Basically an old method, the Fielden Drimeter employs the principle in the form of a parallel plate electrical capacitance through which the web passes; the capacitance forms one arm of an alternating current capacitance bridge, the off-balance signal of which is amplified and indicated on a meter. The general method is described in some detail, and a critical theoretical discussion is given of the application of the method to the indication and control of the moisture content of paper. It is shown that, although the dielectric constant of water is many times that of cellulose, the porous nature of paper and the smallness of the moisture content combine to result in a rather poor sensitivity. However, electrical methods are capable of great sensitivity and accuracy. As used in the Drimeter, the method is quite insensitive because of the fact that nearly all the space between the test capacitor electrodes is air, whereas only a small fraction is occupied by the web. Calculations show that the expected changes in capacitance associated with ordinary changes in moisture content are extremely small, calling for an instrument of extremely great mechanical and electrical stability. The discussion of the Fielden embodiment of the principle of operation brings out the fact that the off-balance signal from an a.c. capacitance bridge is a function of any change in dielectric loss in the test capacitor, as well as of the change in capacitance. The required sensitivity is so high that the mechanical stability of the electrode system must be very great; a change in separation of the electrodes of only 0.000015 inch will produce an apparent change in moisture content of $^1/_2$ unit. Another factor of importance not related to the moisture content is the effect of the variation of the basis weight (and thickness) of the web. The experimental examination of the Drimeter involved a number of effects, including the following. The electrical stability was examined under operating conditions in spaces of controlled atmosphere; the electrical system appears to be fairly stable and adequately sensitive. Tests were conducted with series of plastic films of negligible and appreciable dielectric loss. These tests showed that the instrument responds to nearly pure capacitance changes (as it should), but that dielectric loss also plays an appreciable role. The effect of variations in thickness and basis weight is sufficiently great to result in quite appreciable error in the indication of moisture content. Tests with 16 specially prepared papers under carefully controlled conditions of temperature and relative humidity (the latter at three levels), using an improved technique for "zeroing" the instrument, showed that only a general correlation exists between the moisture content of the papers and the Drimeter reading. In several instances, the Drimeter reading *decreased* when there was an appreciable *increment* in the moisture content, showing that the errors of the method, as employed, and of the instrument, are much too large for the instrument to be regarded as satisfactory for the control of the moisture content of paper. In an additional study of 20 nearly identical sheets of paper, for which the variability of moisture content and of other properties was small, the Drimeter readings covered a range of about 3.6 units; that is, the instrument indicated a substantial variation in moisture content, whereas the actual total spread in the moisture content was only 0.5%. A study of temperature effects showed that a small temperature gradient in the cast aluminum yoke that supports the test capacitor produces error in the Drimeter indication; warming of the yoke near the U-bend by contact with the two hands for one minute produced a change in reading from 20 to 3 units—an apparent change in moisture content of 17%. (This change is for readings before and after contact with the hands so that purely mechanical distortion was avoided.) Although the principle of controlling moisture through its connection with the dielectric constant of a material might, in certain instrumental embodiments of the method, be satisfactory, the Drimeter is judged to be unsatisfactory for the measurement and control of the moisture content of paper.

THE problem of controlling the moisture content of paper during fabrication on the paper machine continues to be one of pressing importance. The electrical resistance method has become increasingly more popular in recent years, probably because of the speed of use of instruments that embody this method and also because the electrical properties of paper depend much more strongly on the moisture content than on other properties. Part I of this report (*1*) presented a study of the electrical resistance method (a.c. and d.c.) and an evaluation of the Hart Moisture Meter and the Moisture Register.

It has occurred to a number of technical men that the extraordinarily high dielectric constant of water ($\epsilon = 81$) suggests the feasibility of measuring the moisture content of grains, foods, paper, and other electrically nonconducting substances by determining the electrical capacitance of a capacitor in which the dielectric is the material under test. One of the earliest of the instruments specifically designed for the control of moisture of paper on the paper machine was the Siccometer, manufactured in Germany by the Siemens and Halske Company (*2–4*). Fielden (*5*) has designed, and is currently producing, an electrical instrument based on this concept. The Fielden Drimeter, although designed primarily for the control of moisture in textiles, has reportedly served with some promise of success in

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the control of moisture content of paper produced in England (*5*). In view of this promise and of the demonstrated accuracy of the basic method as applied in this country to the control of moisture in grains and other foodstuffs, arrangements were made through the kindness of Fielden Electronics, Inc., New York, to study the Drimeter on the American Paper and Pulp Association Instrumentation Program.

THE ELECTRICAL CAPACITANCE METHOD

Before describing the Fielden Drimeter, which is one of many possible embodiments of the electrical capacitance method, it seems desirable to present a brief discussion of the general method. The inherent advantages and disadvantages of the method will then be readily understood, and the underlying reasons for the various experiments in the investigation of the Drimeter should be apparent.

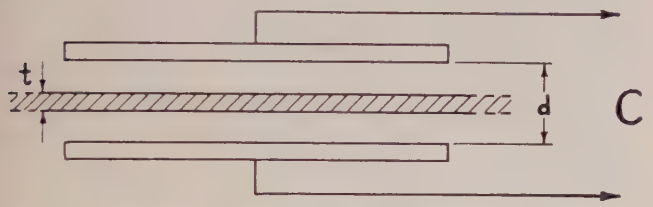

Fig. 1. Two-plate capacitor with dielectric composed of air and sheet material

The paper under test or control (at rest or moving) is made the dielectric of an electrical capacitor (Fig. 1). In its simplest form, the capacitor consists of a pair of plane, parallel metal plates separated a distance d. The paper, of thickness t, only partially fills the space between the plates, so that the capacitance of unit area of the capacitor (in the central zone, remote from the edges of the plates) is given by

$$C = (1.11/4\pi)/[d - t(1 - 1/\epsilon_m)]\mu\mu f/\text{sq. cm.} \qquad (1)$$

The quantity ϵ_m is the dielectric constant of the paper. The definition of this quantity may be given with the help of equation (1). If the paper completely filled the space between the metal plates, so that t would become equal to d, the capacitance would increase in value to $1.11\epsilon_m/4\pi d$ micromicrofarads per unit area. If only air is present between the plates, the capacitance per unit area is $1.11/4\pi d$, within the limits of error with which we are here concerned. (The air and vacuum capacitances differ by a factor of 1.00059; lest the reader be concerned about the influence of the moisture in the air, *per se*, it should be pointed out that the dry air and humid air capacitances differ by an exceedingly small percentage.) It will be seen that the dielectric constant (ϵ_m) is the ratio of the capacitance when the space between the plates is completely filled with the dielectric to that which would be observed or calculated when only air fills the space.

Retaining for the moment the situation in which the space between the plates is filled with paper, the following comments may be made. In the simplest situation, the sheet contains fibers of pure cellulose, pure water, and air. Each component contributes to the observed dielectric constant (ϵ_m) of the paper. If the space were completely filled with water, the dielectric constant would be in the neighborhood of 81. However, the water content of the space occupied by the paper would be typically of the order of 0.04 (corresponding to a moisture content of 5.3% in a paper having a density of 0.75 gram per cc.); this is the crux of the method —although the water content in the space is small, the dielectric constant of water (overlooking the first traces to be sorbed by the cellulose) is so large that its contribution to the observed dielectric constant of the material is measurable.

If all of the space between the plates were completely filled with dry cellulose (a hypothetical case), the dielectric constant would be in the vicinity of 6.1 (*6*). Actually, of course, a substantial fraction of the volume of all papers made from moderately beaten or refined stock is comprised of voids, so that the solid fraction of paper is typically of the order of 0.5; correspondingly, the dielectric constant is substantially less than 6, a typical value being 2.4.

The contribution of a sheet component to the observed dielectric constant does not bear a linear relation to the mass per unit volume of that component. Data on the dielectric constant of dry fiberboard covering a density range from insulation-type board to hardboard showed that the Clausius-Mosotti relation, employed as a purely empirical function without regard for the assumptions on which the relation is based, fitted the experimental observations with good accuracy (*7*). Delevanti and Hansen (*8*) have shown that this function may be similarly employed for handling data on the dielectric constant and loss angle (defect angle) of paper sheets; the function has been similarly employed by Calkins (*9*) and, more recently, Verseput (*10*) has shown that it fits with good accuracy data for sheets prepared from regenerated fibers. In the form employed in these studies, the relation is

$$(\epsilon - 1)/(\epsilon + 2) = k\rho \qquad (2)$$

in which k is a proportionality constant and ρ is the density of the sheet (not to be confused with the density of the solid substance of the fibers, which is usually substantially greater).

For our present purposes, we will assume that water distributed throughout the porous structure of a sheet of paper follows equation (2) and that, also, the dielectric constant of the hypothetical paper composed of only cellulose and water may be calculated from the following equation:

$$(\epsilon_m - 1)/(\epsilon_m + 2) = k_c\rho_c + k_w\rho_w \qquad (3)$$

Taking 6 for the dielectric constant of cellulose at density 1.5 grams per cc., one finds that k_c (the constant for cellulose) is 0.417 cc. per gram; if the dielectric constant of water is 81 at density 1.0 gram per cc., then k_w is 0.964 cc. per gram.

Some notion of the order of sensitivity of the electrical capacitance method may now be obtained. We may calculate the change in dielectric constant corresponding to a change in moisture content from, say, 4.5 to 5.5%. By differentiating equation (3), we find that

$$\delta\epsilon_m = (1/3)(\epsilon_m + 2)^2(k_c\delta\rho_c + k_w\delta\rho_w) \qquad (4)$$

in which $\delta\epsilon_m$ is the change produced in the dielectric constant of the paper occasioned by the increase of

water per unit volume of the sheet ($\delta\rho_w$) and the decrease in the mass of cellulose per unit volume ($\delta\rho_c$) caused by hygroexpansivity of the paper. Estimation of the latter term shows that it is not completely negligible—it is of the order of one-eighth the first term. At a moisture content of 5.0%, the dielectric constant of our hypothetical paper is about 2.62. [In calculating this value with equation (3), one notes that the first term on the right side is 0.313 and the second term is 0.038, showing that, under the supposed conditions of paper density and moisture content, the role played by the cellulose is substantially greater than that of water, in spite of the high dielectric constant of water; it is an unfortunate characteristic of the nature of equation (2)—and of equation (3)—that a slight drop in the mass per unit volume of a substance between the electrodes results in a rapid approach of the observed dielectric constant of the incompletely filled space toward unity.] Applying equation (4) to the estimation of the change in dielectric constant caused by a moisture content change of 1%, we obtain $\delta\epsilon_m = (1/3)(2.62 + 2)^2 (-0.417 \times 0.002 + 0.964 \times 0.0079) = 0.049$, a rather small quantity. Thus, the expectation of the change in dielectric constant of typical paper corresponding to a 1% change in the moisture constant at 5% is not nearly as great as many persons have supposed on the implied assumption of approximate linearity between the density and the contribution to the dielectric constant. This change of 0.049 is about 2% of the dielectric constant.

The smallness of the change in dielectric constant computed above should not, in itself, be too discouraging, for good electrical methods are among the most sensitive and accurate of all methods of measurement. Other factors, however, enter a theoretical discussion of the method as it might actually be applied. If, as assumed by Fielden, paper passed through the space between the electrodes should not make intimate contact with both electrodes (presumably to reduce the chance of a break in the web caused by fouling), the separation d might be substantially greater than t, the thickness of the paper. For example, the Fielden electrodes are separated by about 0.5 cm., and the thickness of typical paper is only 0.01 cm.—a factor of 50. Equation (1) makes allowance for such clearance. Calculations with equation (1) show that an air space between the dielectric and the electrodes has a strong desensitizing effect. The following calculations will bear this out. This effect, it should be noted, is additional to that already discussed in connection with the low level of moisture content of a porous system.

We may calculate the capacitance per unit area of the capacitor for dry paper, for paper containing 3% moisture, and for paper containing 6% moisture (based on total weight). For the purposes of illustrating the orders of magnitude, we neglect the fact that the dielectric constant of the first traces of water sorbed by cellulose is substantially less than the value for free water. Using equations (1) and (3), we find for paper of thickness 0.0035 inch = 0.0089 cm., and density 0.75 when dry:

Dry paper between electrodes: $C = 0.17818$ $\mu\mu$f/sq. cm.
Paper at 3% moisture content: $C = 0.17826$ $\mu\mu$f/sq. cm.
Paper at 6% moisture content: $C = 0.17834$ $\mu\mu$f/sq. cm.

Accordingly, a change in moisture content of 1% should produce a change in capacitance of three parts in 20,000. This is, indeed, a small effect, but it is within the bounds of possibilities with excellent electrical equipment. A question might well be raised, however, concerning the feasibility of a control instrument which is called upon to operate on so small a differential.

Other factors having a bearing on the accuracy of the capacitance method exist. Some of these relate to the properties of the paper and will be treated later. A further equipment factor would seem to be of considerable importance. By what electrical method is the change in capacitance to be measured or utilized in circuitry for control purposes? There are many possibilities. Perhaps the most popular method is that of the alternating current capacitance bridge which, when employed in scientific laboratory work, is capable of great precision and accuracy in the comparison of two capacitors. Because Fielden has developed an application of the bridge method, it is desirable briefly to consider the errors that may be introduced through the use of this method in an electronic system.

An alternating-current capacitance bridge is not balanced unless suitable adjustments have been made upon the reactances *and* phase angles of the arms. A particular illustration is of interest. Suppose that the capacitors consist of air capacitors, and that one of these contains an ideal dielectric (zero loss angle). If the bridge is balanced and one were to replace the ideal dielectric with a slab of a real dielectric having the same thickness and dielectric constant, but also having a finite loss angle, the bridge would not remain in balance. (The bridge could be returned to a balanced condition by, e.g., inserting an appropriate series or parallel resistance with the capacitor in the opposing arm of the bridge.)

Fielden's equipment does not operate on the principle of maintaining the bridge in a completely balanced condition. Where a bridge is employed with an electronic circuit for control purposes, a great simplification of the circuit becomes possible when, after an initial balancing of the bridge, the circuit may operate on the off-balance signal from the bridge. However, an off-balance signal will result when either the loss angle or the dielectric constant (generally both) of the dielectric between the electrodes (paper in the present instance) changes. The dielectric loss in paper is a profound function of the moisture content and, in the region of extremely low moisture content, the loss continues to vary strongly with relative changes in moisture content far beyond the point where the dielectric constant ceases to be influenced (*8*, *9*). In consequence, it is not to be expected that the off-balance signal from a capacitance bridge is a function only of the change in dielectric constant of the paper dielectric in one of the bridge capacitors. The signal will depend also upon the dielectric loss in the paper and, accordingly, will be influenced by any factors other than moisture content which affect the loss angle of the paper.

In view of the required sensitivity in an arrangement like that of Fielden's (a change of 1% moisture content results in a capacitance change of only 3 parts in 20,000), the instrument must possess very great mechanical and electrical stability. For example, any mechanical or thermal action which would change the separation of the capacitor plates by only 7.5×10^{-5} of the normal

separation would produce an objectionable error (0.5% moisture content); in terms of distance of movement, this would amount to only 0.000015 inch.

In view of the nature of equation (1) and the smallness of the change in capacitance of the sensing unit produced by unit change in moisture content, it will be readily appreciated that a change in basis weight (and thickness) of the paper will appear on the meter as a change in moisture content. The magnitude of this effect is discussed later in the report.

DESCRIPTION OF THE FIELDEN DRIMETER

A photograph of the Drimeter is presented in Fig. 2. The apparatus consists of two units: a two-plate

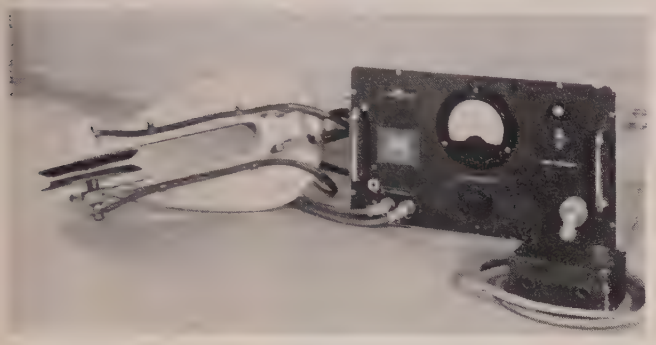

Fig. 2. Fielden Drimeter

capacitor (through which the moving web passes), coupled through coaxial cable to a small metal cabinet which houses the necessary electronic equipment, indicating meter, and controls. As mentioned in the foregoing section, the capacitor in which the dielectric is the moving web of paper is part of an alternating current capacitance bridge; the frequency employed is nominally 500 kc., modulated with a 500-cycle signal of steady amplitude. A simplified schematic diagram is given in Fig. 3. The system includes an r-f bridge, in which two arms are formed by windings of a tuned r-f transformer, one by the "Set Zero" capacitor, and the fourth by the test capacitor. The power supply (Fig. 3a) provides regulated 150 volts to the oscillators and the voltage suppliers and are unregulated voltage to the power amplifier. In operation, the 500-cycle oscillator modulates the output of the 500-kc. oscillator. The bridge is fed with this modulated r-f through the tuned transformer and is balanced by means of the "Set Zero" capacitor with no specimen in place. The bridge output is fed to the tuned grid circuit of the r-f amplifier. With a dry specimen in the test capacitor, the "Set to Dry" control is adjusted until the off-balance signal from the bridge causes a deflection of the meter to a mark on the scale designated as "Dry." This mark is at roughly 24% of full-scale deflection of the meter; there are no scale divisons between the zero and the "Dry" mark, and the scale from this point up to full-scale is graduated in units of moisture content, from zero moisture at the "Dry" mark to 20 units at full-scale.

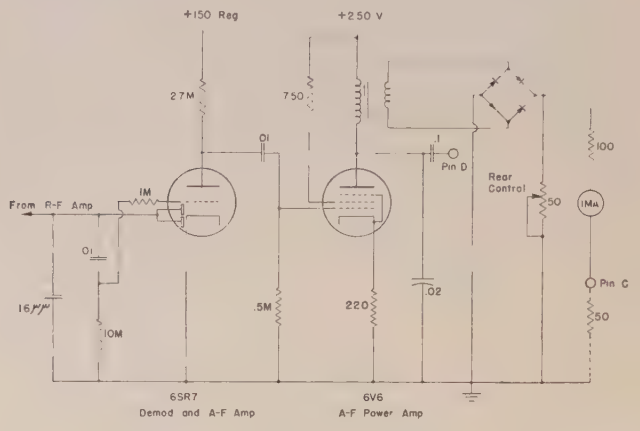

Fig. 3a. Diagrammatic sketch of audio-frequency circuit of Drimeter

The amplified r-f output of the bridge is demodulated by the diode section of the 6SR7 tube, and the resulting a-f signal is amplified by the triode section of that tube. The signal is then passed through the power amplifier to the full-wave dry-disk rectifier, and thence to the one-milliampere meter. An additional meter for remote

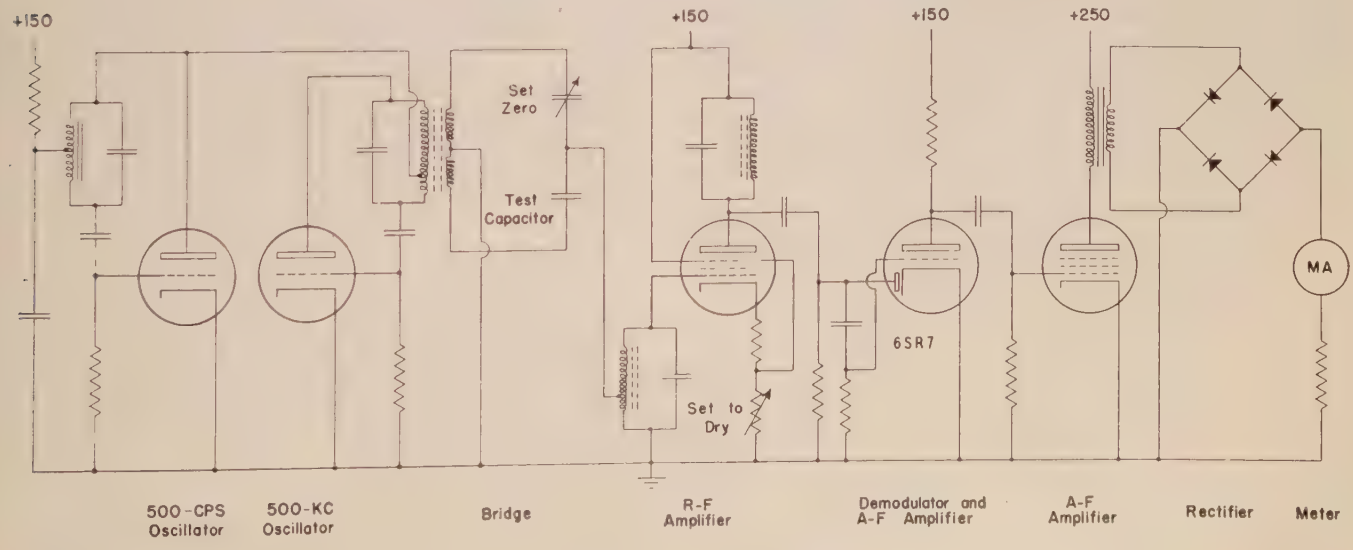

Fig. 3. Simplified schematic diagram of Drimeter

indication may be inserted in place of the 50-ohm resistor in series with the cabinet-mounted meter. The meter on the instrument studied on this program had been calibrated for use with cotton fabric. Calibration for fabrics of other fibers is provided for.

The electrodes are guarded brass disks with a diameter of about 11 cm., mounted at the ends of a heavily constructed metal fork at a separation of about 0.5 cm. In operation, the web is supposed to rest lightly on the electrode marked with a green dot. The instrument is operated on 115-volt, 60-cycle mains.

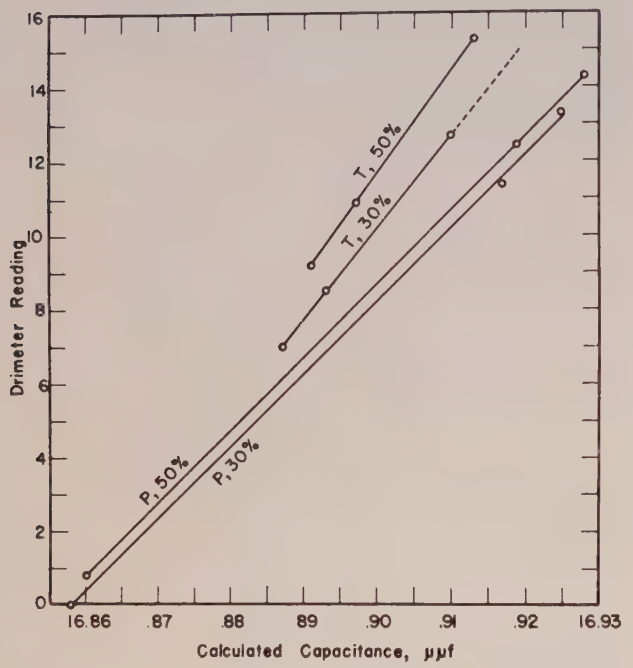

Fig. 4. Drimeter readings versus the calculated capacitance of the test capacitor containing various films of polystyrene (P) and tenite II (T) at 30 and 50% R. H.

A résumé of the adjustment of the instrument is as follows. Using an instrument which has been set for the material under control, the "Set Zero" adjustment is made with nothing but air between the capacitor plates. A dry sheet is then placed between the plates and the "Set to Dry" control (actually a sensitivity control) is adjusted to obtain a deflection to zero moisture (corresponding to about one-fourth of full scale). The instrument is then ready to use, provided, of course, that it had "warmed up" and become steady. Alternatively, after setting the zero (without paper between the electrodes), a sheet of paper of known moisture content is placed between the electrodes and the "Set to Dry" control adjusted to give an indication of the actual moisture content. "Light" and "Heavy" ranges are available; when the control switch is set on "Light," the sensitivity of the instrument is at its maximum value.

EXPERIMENTAL EXAMINATION OF THE DRIMETER

In order that the instrument might be used in various rooms controlled at different temperatures and relative humidities, the Drimeter and related equipment were mounted on a mobile table. In later work, all or part of the equipment was placed in constant temperature, constant humidity cabinets.

Electrical Stability

The input voltage was controlled by means of a variable transformer, and accurately read on a General Electric type P3 voltmeter. A coaxial cable was connected to the plate of the 500-cycle oscillator and led to one plate of a 5-inch oscilloscope. Loading produced by this connection appeared to be negligible. In this manner the 500-cycle frequency could be compared with that of the output of a Hewlett-Packard type 200B audio oscillator by means of Lissajous figures on the screen of the oscilloscope. The amplitude of the 500-cycle oscillations was read from a graph placed over the face of the oscilloscope. A type BC-221-D Signal Corps frequency meter was used to monitor the frequency of the 500-kc. oscillator. The antenna of this meter was loosely coupled with the green-dotted electrode of the test capacitor at the point where the r-f cable is bolted to the electrode. Some loading of the circuit was apparent here but, since the degree of coupling was constant throughout the tests, the loading was not considered to invalidate the data.

Measurements were made in a preliminary study involving sets of films of various thicknesses of polystyrene and "Tenite II," the latter being a cellulose acetate butyrate plastic material which is hygroscopic and "lossy," in contrast to the excellent electrical properties of polystyrene. Because of a finding during this preliminary work concerning the uncertainty in resetting the instrument to zero (to be discussed later), and because the prepared films were not sufficiently uniform for accurate work, the data obtained in a series of measurements at 26, 50, and 89% relative humidity are probably not valid for the purpose of quantitative testing of the significance of the instrumental response. However, the electrical performance of the instrument

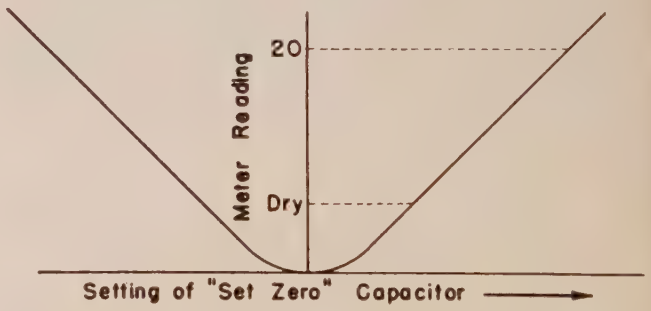

Fig. 5. Schematic representation of the change in Drimeter readings with the setting of the "Set Zero" capacitor

could be tested under conditions of use—in rooms in which the temperature and relative humidity are accurately controlled—and, hence, the data are presented in Table I. The significance of work with polystyrene films of various thicknesses (P-1, P-2, P-3) and Tenite II films of different thicknesses (T-1, T-2, T-3) will be treated later in the report.

The equipment and the plastic films were placed in a 73°F., 26% R. H. room for three days prior to the first tests. After a long warm-up period, the instrument was zeroed with only air between the electrodes, and

then the "Set to Dry" control was adjusted with the thinnest plastic film in place. The deflection could not be brought quite to zero moisture so that a small correction has been applied to all readings. The "Set to Dry" control was taped in its position of maximum gain. Measurements were then made of the frequencies of the two oscillators, and the amplitude of the 500-cycle oscillation, and the Drimeter indications were taken for various supply voltages. This was repeated after three-day conditioning periods in spaces controlled at 73°F., 50% R. H., and at 73° F., 89% R. H.

The data show that the electrical perfomance of the instrument is satisfactory in the different environments, and that the meter readings were fairly stable and reasonably independent of supply voltage fluctuations. The zero was deliberately not reset, so that some notion could be gained about the extent of wander with time and changing conditions. This variation is, in itself, not regarded as serious, because the instrument should be zeroed at reasonably frequent intervals.

The *differences* between the readings obtained with the three polystyrene films under the different environmental conditions were satisfactorily uniform, and fairly independent of supply voltage variations. Polystyrene of electrical grade is known to have low hygroscopicity and very low dielectric loss angle. The increasing differences in the readings of the Drimeter for the Tenite II films as the relative humidity of the environment was increased are undoubtedly to be associated with increasing moisture content and dielectric loss.

The last group of data were obtained after it was noted that lint had collected between the disk electrodes and their respective guards (probably from prior use of the equipment in a textile mill), and the lint had been removed (without disassembling the capacitor). It is interesting to note that removal of the lint augmented the readings for the polystyrene films (low dielectric loss) but did not significantly change the readings for the Tenite II films (relatively high dielectric loss).

Tests of Dependence on Changes in Capacitance and Dielectric Loss

The difficulties of checking an instrument like the Drimeter with regard to its response to quick changes in moisture content will have occurred to the reader. Moreover, it is impossible to make the "Set to Dry" adjustment with a "dry" sheet of paper. Dry paper taken from a desiccator equilibrates very quickly with its new environment, so that any attempt to set the instrument with dry paper will result in a condition which would depend upon the length of time spent by the operator in making the setting. To avoid this difficulty, experiments were conducted with polystyrene films of different thicknesses. Since the moisture content of such films remains very small even at elevated humidities, the thinnest film of a series may be regarded as the "dry" sheet, and thicker films should be equivalent to paper sheets of a common thickness but of different moisture contents, *in so far as capacitance change only is concerned.*

Using auxiliary electrical equipment, the dielectric constant of the polystyrene could be determined and, with the help of equation (1), it was possible to calculate the changes in capacitance that occur when the thinnest film is removed from the Drimeter test capacitor and replaced with a thicker film, and then the second film replaced with a third film. These films were of thicknesses so calculated as to yield changes in capacitance equivalent to moisture content changes within the range of the Drimeter.

The properties of the polystyrene films, and also of three Tenite II films, are given in Table II. The dielectric constant and the loss angle were measured by means of an impedance bridge, and a three-stage amplifier and oscilloscope were used for detection of the bridge balance. Three or four pieces of a film were measured simultaneously in a mechanically loaded capacitor described by Calkins (*9*). Corrections were made for capacitor edge effect, and for spurious capacitance and loss. The frequency employed was 1000 cycles per second. Only a small change in the dielec-

Table I. Stability Tests on the Drimeter

Effects of Variation in Supply Voltage and Relative Humidity

Relative humidity, %			26					50		
500-cycle frequency, cycles/sec.			600					600		
500-cycle amplitude, units			12.0					12.3–12.4		
500-kc. frequency, kc.			405.27–405.23					404.23–404.18		
Drimeter readings, units										
For line voltage of, v.	109	113	117	121	125	109	113	117	121	125
For film P-1	0.0	0.0	0.0	0.0	0.0	0.7	0.5	0.8	0.8	0.9
P-2	[a]	11.3	11.4	11.5	11.7	12.0	12.1	12.4	12.5	12.7
P-3	[a]	13.2	13.3	13.5	13.7	13.9	14.0	14.3	14.6	14.9
T-1	[a]	6.1	7.0	7.1	7.2	[a]	9.1	9.2	9.3	9.4
T-2	[a]	8.4	8.5	8.5	8.7	[a]	10.8	10.9	11.0	11.2
T-3	[a]	12.4	12.6	12.8	13.0	14.9	15.0	15.3	15.5	15.8
Relative humidity, %			89					88		
500-cycle frequency, cycles/sec.			600					600		
500-cycle amplitude, units			12.3					12.3		
500-kc. frequency, kc.			404.15–403.27					406.53–406.48 (Capacitor cleaned)		
Drimeter readings, units										
For line voltage of, v.	109	113	117	121	125	109	113	117	121	125
For film P-1	1.7	1.6	1.5	1.5	1.7	3.2	3.2	3.3	3.4	3.4
P-2	11.6	11.7	11.7	11.7	11.9	12.4	12.5	12.7	13.0	13.3
P-3	15.0	15.2	15.7	16.0	16.4	16.1	16.2	16.9	17.0	17.4
T-1	11.4	11.3	11.5	11.8	12.1	11.3	11.5	11.7	11.9	12.2
T-2	15.8	16.1	16.4	16.8	17.1	15.2	15.5	15.8	16.2	16.8
T-3	20.3	20.5	20.8	21.3	21.8	20.4	20.5	20.8	21.0	21.7

[a] In these instances the indicator went violently off the high end of the scale. Proper behavior was re-established on raising the line voltage to 110 volts.

tric constant is expected when the frequency is changed from this value to 500 kc., and our main interest lies in the dependence of the Drimeter reading on changes in capacitance produced by using different thicknesses of the polystyrene films. The data on loss angle given

Table II. Properties of the Plastic Films

	Film designation					
	P-1	*P-2*	*P-3*	*T-1*	*T-2*	*T-3*
	Dielectric Constant					
32% R. H.	2.08	2.34	2.35	2.96	2.99	3.05
50% R. H.	2.16	2.39	2.42	3.18	3.18	3.21
70% R. H.	2.26	2.38	2.42	3.46	3.51	3.38
	Loss Angle, 0.001 Radian					
32% R. H.	6.2	6.1	6.0	15.8	15.7	16.8
50% R. H.	5.4	6.8	5.7	16.0	15.6	16.7
70% R. H.	6.3	7.6	6.8	23.4	16.7	19.0
	Moisture Content, %					
32% R. H.	0.14	0.08	0.04	0.87	0.87	0.87
50% R. H.	0.18	0.15	0.06	1.5	1.3	1.5
70% R. H.	0.36	0.58	0.14	3.0	2.2	2.6
	Thickness 0.001 cm.					
	3.99	6.65	7.06	5 20	5.51	6.30

in Table II confirm the expectation that the loss angle of the (plasticized) polystyrene is small, and only a slow function of the relative humidity; the films were permitted to equilibrate with changed conditions over a number of days. Also in line with expectation are the increments for the Tenite II in dielectric constant, loss angle, and moisture content, as the relative humidity was raised.

Using equation (1) the capacitance of the Drimeter test capacitor when containing the individual films was calculated. Drimeter readings were obtained after the equipment and the films had come to equilibrium in atmospheres controlled at 73°F. and 30% R. H. and at 73°F. and 50% R. H. The observed Drimeter readings for a line voltage of 117 volts are plotted against the calculated capacitance in Fig. 4. In line with expectation, the curves for the polystyrene films are linear, and the slopes of these lines are of the correct order of magnitude—that is, the design sensitivity of the Drimeter appears to be satisfactory.

The two curves for the set of polystyrene films at 30% and 50% R. H. should be superposed. However, an examination of the dependence of the Drimeter zero setting on relative humidity (Table III) showed that the balance point changes with relative humidity by at least enough to account for the difference between these curves.

Of more interest are the pair of curves for the set of Tenite II films at 30% and 50% R. H. These lie substantially above the curves for the polystyrene films, and the discrepancy increases with increasing moisture content. It should be emphasized that changes in capacitance associated with changes in dielectric constant caused by changes in moisture content have been included in the calculations and that, if the Drimeter readings were dependent only on the dielectric constant, the data for the Tenite II films should have fallen on the curves established by the polystyrene films. As discussed earlier, it is expected that the off-balance signal from a capacitance bridge should depend upon changes in dielectric loss as well as on changes in dielectric constant of a bridge arm capacitor. It is concluded, therefore, from the higher Drimeter readings obtained with the Tenite II films that the response of the Drimeter depends upon the dielectric loss of the test specimen, in addition to the dielectric constant.

Dependence on Thickness of Homogeneous Films

It should be stressed that the large, over-all changes in capacitance and Drimeter reading occurring in the data for the polystyrene and Tenite II films (Fig. 4) were associated simply with changes in thickness of the films. Fielden recognizes that changes in the weight and thickness of fabric and paper will produce a change in instrumental response, but asserts that the change associated with a modification of the moisture content is much the greater. A question is here raised as to whether considerations of the kind previously discussed in connection with equations (1) and (2)—especially with regard to the desensitizing aspects of the porosity of a sheet—have been made by Fielden. In the case of the polystyrene films, in which the moisture content was almost negligible, one has an almost pure measure of the effect of thickness variation. It is noted that a change in thickness from about 0.004 to 0.007 cm. caused a change in Drimeter reading of 13 units. In this range, the response is linear, so that a change in thickness and of basis weight of 6%, which is commonly encountered in the production of paper, would produce a change in Drimeter response of one unit. It is obvious that this apparent change in moisture content of one unit is completely in error. With regard to the Tenite II films, in which the moisture content was appreciable, the Drimeter reading for T-3 at 30% R. H. was 12.6 units, whereas the reading for T-1 at 50% R. H. was 9.1 units; however, the moisture contents were, respectively, 0.9% and 1.5%. It is realized that the Drimeter was not calibrated for Tenite II films; but we are here concerned with an *inversion* of serious proportions. In view of the earlier discussion centering on equation (2), the situation might well be worse in a porous, even though more hygroscopic, material such as paper.

Test of the Drimeter with Specially Prepared Papers

In an investigation of an instrument like the Drimeter, it is, of course, very desirable to test the equipment with paper.

Prior to the performance of this important test, it was found desirable to improve the technique of adjusting the Drimeter. In earlier phases of the work it was observed that the zero adjustment was insufficiently precise for readings in the normal range. It is to be emphasized that this would not necessarily be a source of difficulty in the normal use of the Drimeter; it is a source of error in an examination of the instrument. The difficulty may be readily understood by reference to Fig. 5, which presents the variation in meter reading (indication of degree of unbalance) with with the setting of the "Set Zero" capacitor. The normal range exists in a fairly linear portion of the curve where the slope (sensitivity) is high. At and near the minimum or zero setting, the slope is zero or very small; the zero setting, made within the limits of detectability, involves uncertainties which correspond to quite large discrepancies on the indicating meter in the range between "Dry" and 20 units.

The technique employed in zeroing the instrument was to adjust to equal meter readings on both sides of the minimum, and then to set the adjusting capacitor to the point halfway between the two settings. The "Set Zero" shaft was extended and attached to a 4-inch vernier dial. Even with this arrangement, however, the required change in the setting of the capacitor was so small that insufficient accuracy could be obtained. A simple modification was made to improve the accuracy; a fixed capacitor in parallel with the "Set Zero" variable capacitor was disconnected and a variable capacitor (already present in the compartment, but not connected) was connected in its place. (This added capacitance was sufficiently great that a low-loss series condenser was added.) Thus coarse and fine adjustments by means of the two variable capacitors were provided.

After the Drimeter had conditioned for several days at 73°F. and 30% R. H., the following readings were taken in the setting of the zero.

Meter deflection to	*"Set Zero"*	*Dial*	*Zero*
"Dry"	44.1	61.1	52.6
5 units	42.3	62.7	52.5
10 units	41.0	64.3	52.65

These zero settings cover the following actual meter deflections (above the meter zero): 6.3, 11.3, and 16.3 units. Thus, within this fairly large relative range, and within the accuracy of setting, the curve shown in Fig. 5 is symmetrical, and the improved method for setting the zero is applicable.

One of the greatest difficulties in investigative work of this nature is that of providing a standard whose effect, when inserted in the Drimeter capacitor, will be the same under all conditions of relative humidity. A polystyrene film would probably be suitable for low and moderate relative humidities (see Table II). A 0.0025-inch sheet of "Teflon" was chosen as a reference standard because of its excellent electrical characteristics and very low sorption of moisture. Moisture sorption tests conducted at 73°F. and at 32, 50, and 70% R. H. yielded the following moisture contents: 0.07, 0.07, and 0.36%, respectively. It seems probable that a significant portion of this sorption is that occurring on the surfaces of the Teflon. The film of Teflon was mounted on a frame comprised of thin brass rod, the latter being sufficiently large that its proximity to the test capacitor had no influence on the Drimeter indication. This film was employed as a standard and was used to set the meter to the deflection marked as "Dry" in all the work that follows.

At 30% R. H. the zero was found and set at 52.6 divisions on the special vernier dial. The meter was then set to "Dry" deflection with the Teflon film in place. The film was removed, and several determinations were made of the amount the "Set Zero" control could be moved before a discernible deflection of the meter occurred. The averages of the readings for the two directions away from the zero setting were 47.90 and 56.83, or a total spread of 8.93 divisions. Judging from this result, it seems conservative to say that the instrument could be zeroed to within ±3 divisions of the true zero in the absence of the special zeroing arrangement. The relationship between divisions of the vernier dial and meter reading can be seen from the fact that the vernier dial was turned through 3.2 divisions corresponding to a change in meter reading

Table III. Dependence of Zero Setting on Relative Humidity, 73°F.

Drimeter reading	—30% R. H.—		—50% R. H.—		—70% R. H.—	
"Dry"	44.1	61.1	43.3	60.7	38.6	56.8
5 Units	42.3	62.7	41.5	62.4	36.8	58.6
10 Units	41.0	64.3	40.0	63.9	35.1	60.1
"Dry" to 10	3.1	3.2	3.3	3.2	3.5	3.3
Zero	52.6		52.0		47.7	

from "Dry" to 10 units. This means that the zero cannot be set to within ±10 units on the Drimeter indicator, although the method of setting the instrument to "Dry" tends to minimize this error. The "Set to Dry" adjustment is really one of sensitivity and, when made with a dry specimen in place, ensures that the response is correct when in the vicinity of the "Dry" deflection; but, to the extent that the gain is different from its proper value because of the error in zeroing, the instrument would be in error at deflections substantially away from the "Dry" deflection.

With the instrument in equilibrium first at 73°F., 30% R. H., then at 73°F., 50% R. H., and finally at 73°F., 70% R. H., the "Set to Dry" control was turned to yield maximum amplifier gain, and zero readings were taken by the technique described above. The data obtained are presented in Table III. The first column gives the meter deflection employed in obtaining the zero. Each pair of numbers under a stated relative humidity (e.g., 44.1 and 61.1) are the vernier dial readings below and above the zero, for the meter deflection given in the first column; the average value of a pair of numbers is the setting corresponding to the true zero. The means of these averages for the three relative humidities are given in the bottom row of the table. It will be seen that the various effects of humidity on the instrumental factors combine in such manner as to cause the zero setting to change by 0.6 and 4.3 divisions, respectively, for the changes in relative humidity, 30 to 50%, and 50 to 70%. The data presented in the next to last row of the table show that one division of the vernier dial is equivalent to 3 Drimeter units; accordingly, the drift associated with the humidity changes could be as large as 13 Drimeter units, if the instrument were not regularly checked and adjusted.

Prior to the taking of the observations presented in Table III, it had been noticed that the zero setting drifted badly at 90% R. H. when only the test capacitor was exposed to that condition. The capacitor plates are insulated from the cast aluminum yoke by means of washers and tubes that appear to be made from a laminated paper-base plastic. Without doubt, this plastic sorbed moisture and became increasingly "lossy" at the higher relative humidities, thus unbalancing the bridge.

Following the improvements in setting the zero and standardizing the instrument through the use of a Teflon sheet, the test of the instrument with paper was made. A series of handsheets was made in the Institute pulping laboratory according to the specifications used in the preparation of handsheets for the investigations reported in Part I of this report (*1*). The designa-

Table IV. Description of the Handsheets

Paper	Variable	Fiber furnish, %						Beating time, min.	Schopper freeness, ml.	pH		Alum, %	Size, %	Basis weight (25 × 40—500), lb. (oven dry)	Remarks
		Bleached sulphite	Unbleached sulphite	Groundwood	Unbleached kraft	Soda	Rag			In beater	In mold				
100	Standard	100	..	..	..	..	..	32	575	7.7	..	..	..	42.7	
101	Furnish	..	15	85	..	..	..	1	345	..	..	..	..	42.7	
102	Furnish	..	..	..	100	..	..	36	730	..	..	..	..	42.7	
103	Furnish	35	..	..	..	65	..	33	430	..	..	..	..	42.7	
104	Furnish	..	..	..	..	..	100	37	350	..	..	..	..	42.7	
105	Beating	100	..	..	..	..	..	20	735	7.8	..	..	..	42.7	
106	Beating	100	..	..	..	..	..	43	400	7.7	..	..	..	42.7	
107	Size	100	..	..	..	..	..	27	690	4.6	5.3	2	3	42.7	
108	Density	100	..	..	..	..	..	32	600	7.7	..	..	..	42.7	One pressing
109	Basis weight	100	..	..	..	..	..	32	600	7.7	..	..	..	128	
110	pH	100	..	..	..	..	..	33	600	4.15	6.5	7	..	42.7	
111	pH	100	..	..	..	..	..	33	600	3.7	5.25	35	..	42.7	
112	Ash	100	..	..	..	..	..	33	580	7.8	..	..	..	42.7	35% clay added
113	Dye, acid	100	..	..	..	..	..	33	610	4.6	4.4	2	3	42.7	[a]
114	Dye, basic	100	..	..	..	..	..	33	610	..	..	2	3	42.7	[b]
115	Dye, direct	100	..	..	..	..	..	33	600	..	..	2	3	42.7	[c]

[a] 8 lb./ton Auramine yellow PEX, conc.
[b] 8 lb./ton Auramine, conc.
[c] 4 lb./ton Red 8BL.
Consistency 1.57%; No. 3 Valley beater, 5500-gram bedplate load; British sheet mold.

tions, furnishes, etc., for these handsheets are given in Table IV.

The handsheets were wet pressed twice, ten at a time, each sheet between blotters, with the wire side of the blotters in contact with the sheets. After the first pressing at 50 p.s.i.g., one blotter was removed from each sheet and replaced with a new blotter. The sheets were again pressed, then dried between blotters for 5.5 minutes on a small steam drier in which the steam pressure was 9 p.s.i.g. In the preparation of Paper 108, only one pressing was used, as this paper was intended to have a low density.

In order that the sheets could be maintained in a flat condition, each sheet was furnished with peripheral tabs, by means of which the sheet was sewed to an 8-inch brass ring. It was established that the proximity of the brass ring introduced no error. A simple support for the ring was constructed, so that the relative position of the test specimen in the capacitor was correct and reproducible.

After preconditioning at a low relative humidity, the sheets and equipment were placed in a room in which the temperature and relative humidity were controlled to 73°F. and 30% R. H., and tests were made after a long period of conditioning and a warm-up of the instrument of at least one hour (20 minutes warm-up is recommended, and is evidently the minimum). Three specimens of each of the 16 paper types were prepared for support in the brass rings. Other sheets of the same types were conditioned simultaneously and placed in weighing bottles at the time the companion sheets were tested in the Drimeter. Thus, the same sheets were tested in the Drimeter at each relative humidity, and errors associated with imperfect formation, basis weight variation and other sheet imperfections should have been virtually nil. In all cases the true zero was obtained by the improved technique previously described; the Teflon sheet was used to set the meter to "Dry." Following the work at 30% R. H., the tests were continued after proper conditioning times at 73°F., 50% R. H. and 73°F., 70% R. H. It should be noted that, in all this testing, conditions were fairly ideal; the temperature was uniform, the paper specimens and the equipment were in equilibrium with the environment, and moisture contents were determined through accurate weighings on an analytical balance and oven drying to constant weight; there was no need to assume that desiccated paper would remain dry during the time required to remove it from a desiccator and perform the "Set to Dry" adjustment; nor was it necessary to assume that paper taken from one environment and tested in another would retain

Table V. Drimeter Readings and Moisture Contents of Papers Conditioned at Three Relative Humidities

Paper	30% R. H.		50% R. H.		70% R. H.	
	Reading, units	Moisture, %	Reading, units	Moisture, %	Reading, units	Moisture, %
100	−1.2	5.41	−1.0	6.59	0.4	9.06
101	8.9	6.33	12.8	7.86	14.2	10.87
102	0.7	5.99	3.3	7.32	5.4	10.00
103	2.8	5.49	4.8	6.96	5.9	9.01
104	2.1	4.97	3.7	6.04	4.3	8.17
105	3.7	5.29	5.8	6.98	7.5	8.88
106	−0.8	5.07	0.0	6.84	1.6	9.03
107	2.3	5.15	4.3	6.95	5.8	8.87
108	2.6	5.19	1.8	6.65	3.0	9.04
109	5.9[a]	5.19	6.8[a]	6.68	5.3[a]	8.86
110	1.4	5.54	3.3	7.19	1.6	9.45
111	2.2	6.11	3.0	7.76	3.9	10.05
112	0.3	4.74	0.9	6.23	0.3	7.94
113	0.8	5.45	1.3	6.86	2.8	8.79
114	2.3	5.33	3.5	6.84	3.5	8.83
115	2.8	5.59	3.9	6.98	4.0	8.90

[a] Readings taken in "Heavy" range.

the former moisture content. The nearly ideal conditions of testing should be kept in mind while studying the results presented in Table V and in Fig. 6.

It will be seen that there is only a general correlation between the Drimeter reading and the moisture content. A statistical study was not made because inspection of the data reveals readily that the Drimeter in its

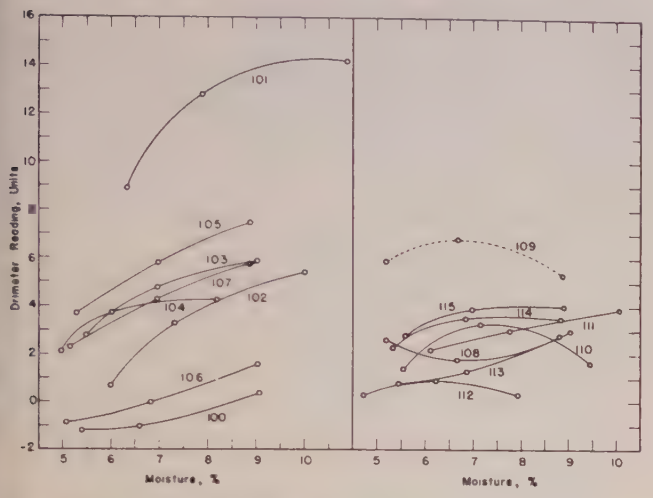

Fig. 6. Drimeter readings for various paper specimens versus their directly measured moisture contents

present form cannot function satisfactorily for the indication of the moisture content of paper. The most revealing indication of gross error is the fact that, *in four cases, the reading of the Drimeter actually decreased significantly when the moisture content increased in relatively large steps* (Papers 108, 109, 110, and 112). In four other cases, there are regions in which the Drimeter reading scarcely changed when the moisture content underwent fairly large increments (Papers 100, 104, 114, and 115).

During the course of the work with the handsheets, the operator made the observation that frequently there was a considerable variation in the reading of the Drimeter in going from one handsheet to another of the same type having almost exactly the same past history. This observation led to the testing of 20 sheets of Paper 100. These were calipered, tested on the Drimeter, and weighed for basis weight and moisture content, after a long period of conditioning at 73°F. and 50% R. H. The results are presented in Table VI and Fig. 7.

These 20 sheets of Paper 100 were not preconditioned in the same manner as those previously employed so that, because of the hysteresis effect, the average moisture content was somewhat higher than that of the sheets in the earlier series. Omitting the Drimeter readings for the moment, it may be said that the data covered small ranges; in such circumstances, when the errors of the measuring methods are not greatly less than the spread of values, poor correlation or no correlation at all between various quantities is to be expected. The points present a typical representation of essentially zero correlation: an almost random distribution of points in rectangular zones parallel to the frame of reference. With regard to the Drimeter readings, it will be observed that, in spite of the small spreads in basis weight, caliper, moisture content, and density, the range covered by the Drimeter readings was about 3.6 units. In contrast, the total spread in actual moisture content was less than 0.5%. If this had been paper running on a paper machine (at essentially constant moisture content), the instrument would have misled the machine tenders into believing that the variation was more than ±1% moisture. In a number of mills, variability of moisture content of this order is regarded fairly seriously.

Temperature Effects

The work described thus far has been conducted at uniform temperature. In a paper mill the temperature is not constant at any point in or near a paper machine. It has already been pointed out that the test capacitor of the Drimeter must be mechanically very stable; it will be recalled that, by computation, a variation in the distance of separation of the test capacitor plates of only 0.000015 inch would cause an apparent change in the moisture content of the paper under test of about 0.5%. Conceivably, temperature effects could be operable in two ways. First, at *steady* temperatures of different values, the cast aluminum yoke that supports the capacitor plates might assume slightly different shapes because of inhomogeneities and residual stresses.

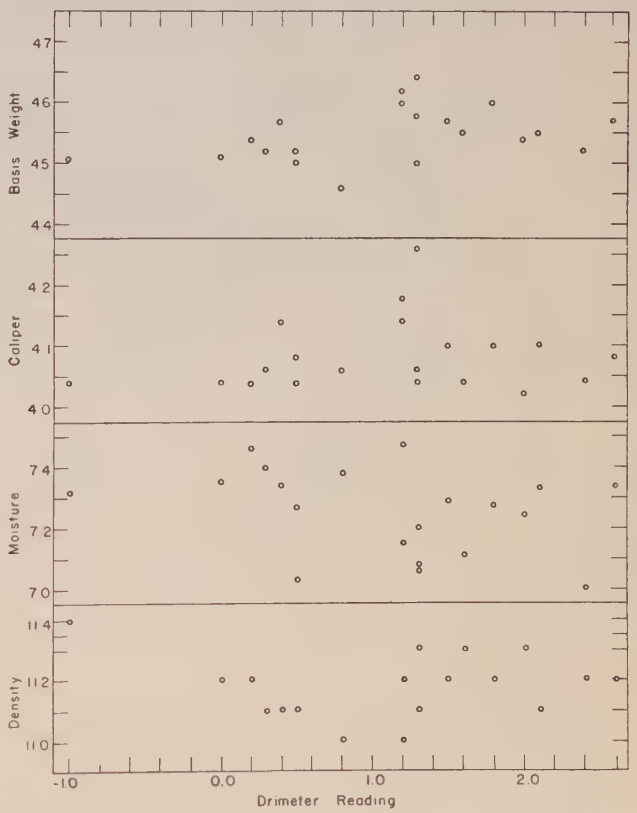

Fig. 7. Drimeter readings for a series of 20 similar paper specimens versus certain physical properties

Second, under conditions of *changing* temperatures, differential expansion (or contraction) of the metal might produce a slight warping of the yoke.

In the testing of the first possible influence of temperature, the whole equipment and a Teflon sheet were placed in a cabinet in which the temperature was controlled at three different levels. The "Set to Dry"

adjustment was turned to maximum gain and, at each temperature, ample time was permitted for thermal equilibration before readings were taken. The work was then repeated with the electronic system and indicator outside of the cabinet, in a conditioned room at 73°F. and 50% R. H. The results of this experiment are given in Table VII.

Table VI. Characteristics of Twenty Sheets of Paper 100

Sheet	*Caliper, 0.001 in.*	*Basis weight (25 × 40—500), lb./ream*	*Density*[a]	*Moisture, %*	*Drimeter reading*
A	4.06	44.6	11.0	7.38	0.8
B	4.04	45.1	11.2	7.35	0.0
C	4.14	45.7	11.1	7.34	0.4
D	4.04	45.1	11.4	7.32	−1.0
E	4.04	45.4	11.2	7.46	0.2
F	4.10	46.0	11.2	7.27	1.8
G	4.06	45.8	11.3	7.20	1.3
H	4.10	45.7	11.2	7.29	1.5
I	4.10	45.5	11.1	7.33	2.1
J	4.04	45.0	11.1	7.27	0.5
K	4.14	46.2	11.2	7.15	1.2
L	4.02	45.4	11.3	7.24	2.0
M	4.06	45.2	11.1	7.40	0.3
N	4.08	45.7	11.2	7.34	2.6
O	4.18	46.0	11.0	7.48	1.2
P	4.08	45.2	11.1	7.03	0.5
Q	4.04	45.2	11.2	7.00	2.4
R	4.04	45.0	11.1	7.08	1.3
S	4.04	45.5	11.3	7.11	1.6
T	4.26	46.4	10.9	7.06	1.3

[a] Density = Basis weight/caliper.

The zero drift was not large and, in practice (at any steady temperature), regular checking of the instrument would take care of the effect. The readings obtained with the Teflon in the test capacitor should not have varied; it is odd that the variation proceeded in the opposite direction when the electronic system was removed from the cabinet.

Table VII. The Influence on Drimeter Readings of Steady Temperature at Different Levels

Temp., °F.	*Relative humidity, %*	*Zero, div.*	*20 to "Dry"*[a]	*Reading, Teflon*	
73	46	50.3	5.6	8.0	Whole equipment
88	35	50.4	5.7	10.0	in cabinet
99	26	51.0	5.9	10.5	
69	56	50.1	5.8	8.0	Electronic system
79	38	50.5	5.7	7.3	outside cabinet
90	31	51.7	5.7	6.7	

[a] Change in the vernier dial reading on the zero adjuster corresponding to a change in meter deflection from 20 to "Dry."

A simple, qualitative observation showed that the effect upon the Drimeter of *changing* temperature of the cast aluminum yoke was very strong. With the instrument in steady operation after a suitable warm-up period, the operator laid his hands upon the top arm of the yoke, in the region near the U-bend, for 1 minute. The hands were removed before a reading was noted, because the great sensitivity of the system to mechanical forces was recognized. Before the warming with the hands, the Drimeter reading was 20 units; after warming, the reading was 3 units—an apparent change in moisture content of 17%. The indicator returned toward 20 units as thermal equilibrium became re-established. It goes without saying that a very small temperature gradient in the yoke, associated with a small rate of heat transfer through the aluminum, would cause appreciable error. In the vicinity of a paper machine, temperature gradients, associated with radiation and the movement of air, are quite strong—probably of sufficient strength in some locations to produce substantial error in an application of the Drimeter to a paper machine.

CONCLUSION

Although the principle of controlling moisture through its connection with the dielectric constant of a material might, in certain instrumental embodiments of the method, be satisfactory, the Drimeter is judged to be unsatisfactory for the measurement and control of the moisture content of paper.

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Paper and Packaging Specifications

Federal Specifications

The following information on Federal Specifications of recent issue comes from Robert B. Hobbs, National Bureau of Standards, Member of the Technical Committee on Paper and Paper Products, Federal Specifications Board.

UU-P-388e (Amendment), Paper, Mimeograph; Price of the Specification and amendment, 5 cents

This amendment does not affect the requirements for the mimeograph paper itself in any way, but merely clarifies and brings up to date some of the packaging and packing requirements applicable to Army, Navy, and Air Force purchases.

UU-B-71b (Revision), Bands, Paper, Shirt, (Laundry); Price, 5 cents

This revision supersedes UU-B-71, dated March, 1933, and Interim Federal Specification UU-B-71a, dated Oct. 26, 1951. The minimum length of the band is reduced from 20 to 19 in. The bands shall be gummed for a distance of not less than $1^1/_2$ in. from the end, or shall have a strip of gummed paper affixed to one end and extending not less than $^3/_4$ in. from the end. A tolerance of ±0.8 lb. is added for the basis weight of 16 lbs. (17 × 22—500). A sample for testing shall consist of not less than 10 bands, and it is required that bands be packed in bundles of 1000. The requirement for ash content is deleted.

(Copies of Federal Specifications and the Federal Specifications Index may be obtained on application accompanied by postal note, money order, coupon, or cash to the Superintendent of Documents, Government Printing Office, Washington 25, D. C. The price of the Index is 40 cents.)

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Seventh Engineering Conference, Morrison Hotel, Chicago, Ill., October 13–16, 1952.

Fibrous Agricultural Residues Conference, Northern Regional Research Laboratory, Peoria, Ill., October 27–28, 1952 (Rooms at Pere Marquette Hotel).

Plastics Conference, College of Forestry, State University of New York, Syracuse, N. Y., November 5–6, 1952.

Alkaline Pulping Conference, Battle House, Mobile, Ala., November 12–14, 1952.

Corrugated Containers Conference, Schroeder Hotel, Milwaukee, Wis., November 20–21, 1952.

Annual Meeting, Commodore Hotel, New York, N. Y. February 16–19, 1953.

LOCAL SECTIONS

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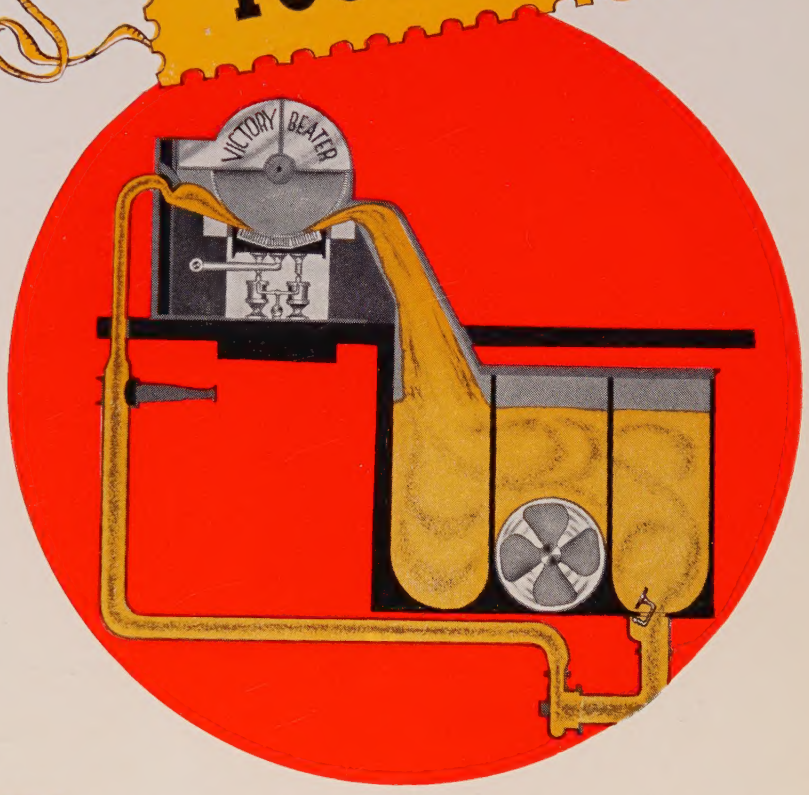

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